ABSTRACT

Caisley, Lena Louise. Nitrogen Fertilization Effects on Soil pH and Calcium Fertilization on Growth and Form of Loblolly Pine. (Under the direction of Dr. Rachel L. Cook).

Loblolly pine (Pinus taeda) is the most important commercially grown pine species in the southeastern United States. Decades have been spent researching different silviculture practices to optimize growth of loblolly pine. The most important practice employed in the management of loblolly pine is fertilization. Nitrogen (N) and Phosphorus (P) are two most common growth-limiting factors of loblolly pine. Little research has been conducted to determine the consequences of long-term fertilization on forest soils.

Soil is a nonrenewable resource; due to the thousands of years it takes to form. Protecting and caring for the soil is a vital step in optimizing growth of all plants. Many N fertilizers acidify the soil, lowering soil pH, which influences many soil factors including nutrient availability. While loblolly pine is adapted to grow in a pH range of 4.5-6, lower than most agronomic crops, little is known about the effects of long-term acidification due to increased nutrient inputs.

Agronomic crops, such as corn, are fertilized annually with N as well but to counteract the acidifying effects of N, calcium fertilizer (Ca) in the form of calcium carbonate (lime) is also applied to raise the pH. In South America, it is common practice to apply agricultural lime to a harvested loblolly stand before the next rotation is established to raise soil pH and ensure good nutrient availability, however Ca fertilization is not common practice in the southeastern US on loblolly pine stands. In addition to controlling pH, Ca in lime is an important nutrient, as it reinforces the structure of cell walls and is vital to the formation of wood. Imbalances of Ca with other nutrients may lead to deformities such as stem sinuosity.

To address these issues, we examined loblolly pine stands with juvenile fertilization treatments at sites located throughout the southeastern US to examine the effects of increasing

rates of N application on soil pH across a variety of different soil textures and drainage classes. Another study was established to examine the effects on growth response and stem sinuosity with the addition Ca fertilization on loblolly pines grown on deep organic soils where stem deformity is common.

Finer textured soils showed soil pH was lowered significantly down to a meter by greater rates of urea fertilizer. Coarser textured soils did not show significant soil acidification. Poorly and well-drained stands both experienced soil acidification down to 30 cm, but poorly drained stands had soil pH lowered down to a meter. Base saturation was lowered by the two highest N treatments, but CEC of the soil remained unaltered. No adverse growth affects were detected at lower pH levels. It is not yet known how these results will translate to the next rotation of loblolly pine but decreased base saturation of the soil may indicate future problems.
Loblolly pines do not require N additions when grown in organic soil, as organic soils have an abundance of N already. Effects of Ca fertilization on organic soils are not well documented, but that is about to change. Three-year results have shown that Ca fertilizer on loblolly pines on organic soil increases growth response in diameter and volume and shows an improvement in sinuosity. There has been some genetic interaction with the treatments in regard to height, DBH and volume. We hypothesize that our initial treatments of 400 kg ha^{-1} Ca as Calciprill, and both 800 kg ha^{-1} Ca as Calciprill and dolomitic limestone were too small to see a large treatment effect. Hurricane damage likely contributed to differing results from the two field sites, except for height to live crown and sinuosity ratings which were similar. To continue and expand the study, three new sites were established that will continue into the future.

Nitrogen Fertilization Effects on Soil pH and Calcium Fertilization on Growth and Form of Loblolly Pine

by
Lena Louise Caisley

A thesis submitted to the Graduate Faculty of
North Carolina State University
in partial fulfillment of the
requirements for the degree of
Master of Science

Forestry and Environmental Resources

Raleigh, North Carolina
2021

APPROVED BY:

_____	_____
Dr. Rachel L. Cook	Dr. Alex Woodley
Committee Chair	Committee Member
_____	
Dr. Luke Gatiboni	
Committee Member	

DEDICATION

To my parents and boyfriend

BIOGRAPHY

Lena Caisley grew up in Danvers, Illinois where her parents taught her the value of discipline and hard work. She attended the University of Kentucky where she graduated with a Bachelor of Science in Forestry. She pursued forestry because of her love of the outdoors, science, and to be a good steward of the earth. During the summer of her junior year of college, Lena was given the opportunity to be an intern at Molpus in Bay Minette, AL. During that summer, she learned a great deal about southern pine plantations and the industry as a whole. At the end of that summer, she attended a conference hosted by the Forestry Productivity Co-op (FPC). After she completed her senior year at UK, she went on to pursue a Master of Science at NC State University, researching fertilization of loblolly pines.

ACKNOWLEDGMENTS

I have been encouraged, sustained, and inspired by my parents, my boyfriend, my sister, my friends, my advisor and lab group. Without their enduring support, none of this would have been possible. I owe every one of you the world's biggest thank you.

To my parents, thank you for every sacrifice you made for me. Thank you for your constant encouragement to pursue all of my dreams and making it possible. For the thousands of miles you have driven to Lexington, KY, Bay Minette, AL and Raleigh, NC, thank you. For every call where I thought the world was falling apart and you were both there to reassure me it wasn't, thank you. Thank you for teaching me discipline, raising me to be a hardheaded feminist and that anything is possible with a little hard work. I will never be able to repay either of you for how much you have done for me and I just want you to know how much I appreciate and love you both.

To my boyfriend, I have not been the easiest to live with during this whole process but I thank you so much for sticking with me through it. You have kept me sane and been my rock. Thank you for constantly being my cheerleader and making me smile. You inspire me to keep going when the going gets tough. I love you and you are my number one.

To my sister, thank you for all of the funny FaceTime's and gif texts when I was extremely stressed. Thank you for the constant inspiration of your undergraduate work. You are truly amazing, amazing and so smart! Love you Bean!

Without my friends, my mental state would be in even worse tatters than it is now! Thank you all for constantly checking in on me and believing in me. Thank you for all the calls and messages.

I would also like to thank my advisor, Dr. Cook, for giving me the opportunity to work on this project and for believing in me. I am beyond thankful for your mentorship. You are an inspiration to myself and all women in forestry.

To the most amazing lab group ever…Thank you! I have learned so much from every single one of you. Thank you for the help with fieldwork, arc gis, and the reminder that it is okay to take a break and care for yourself. I hope we get to work together again someday!

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

CHAPTER 1

Nitrogen Fertilization Effects on Soil pH in Loblolly Pine Stands

Introduction

Loblolly pine (*Pinus taeda L.)* is grown in the southeastern United States, across many different soil types and terrains, and is one of the most economically important trees in the world today. Demand for loblolly pine is projected to grow 30-40% with increasing population, while production is anticipated to continue to shift away from privately owned natural forests to corporately owned pine plantations (Allen et al. 2005). Products from loblolly pine include pulpwood, plywood, solid wood, poles and a wide range of other different products. To meet the demand for wood products, private sector companies and landowners grow loblolly pines commercially in plantations and heavily manage the stands to achieve optimal growth (Fox et al. 2007a). Loblolly pine stands are first thinned around fifteen years old and harvested around twenty-five to thirty years.

Nitrogen (N) is an essential nutrient and most often limiting to all major processes and development of plants. In loblolly pine in the southeastern US, nitrogen is typically the most limiting factor, followed by phosphorus (P). P fertilization commonly occurs at or shortly after stand establishment. N+P fertilization of loblolly pine stands generally occurs at mid- rotation (Fox et al. 2007a). Fertilization of loblolly pine stands has been common practice in the southeastern US since 1999 (Albaugh et al. 2007). Fertilization of a stand can move stands from low quality timber products (chip and saw) to higher valued timber products such as saw timber, much faster than stand that had not been fertilized (Albaugh et al. 2019). Out of all silviculture tools, fertilization has the greatest effect on loblolly pine growth shortening rotation lengths (Carlson et al. 2008).

Loblolly pine fertilized with N photosynthesize more than untreated trees (Gough et al., 2004) and have greater leaf area index (LAI) (Albaugh et al., 1998). Loblolly pine generally responds to N+P in diameter height and overall volume growth (Carlson et al. 2013). However, growth response to N fertilization is relatively short lived (approximately 8 years), while the response to P fertilization is persistent for many years post fertilization (Fox et al. 2007b).

Urea ($CO(NH_2)_2$) is the most commonly used source of nitrogen fertilizer in forest plantations (Albaugh et al. 2019) and has less acidifying potential than other N fertilizers such as monoammonium phosphate and ammonium sulfate (Adams 1984). Before the plant can take up the nitrogen from the urea fertilizer it is first hydrolyzed to ammonia (NH_3), then reacts with soil water to form ammonium (NH_4^+). As soil microorganisms transform ammonia to nitrate (NO_3^-), ions hydrogen (H^+) are released. This process causes acidification of the soil when the N inputs are greater than the ability of the soil microorganisms and organic matter to buffer the acid cation (Barak et al. 1997). At lower pH levels, microorganisms, such as ectomycorrhizal fungi which are important for nutrient acquisition for loblolly pine may have their activity reduced (Zhao et al. 2011).

Loblolly pine typically grows well in soil pH ranging from 4.5 to 6, which is considered to be very strongly to moderately acidic (USDA NRCS 1998). However, as fertilization of plantation pine has become more commonplace, soil acidification has the potential to lead to nutrient limitations. Many plant macronutrients are known to be less available at lower pH's in agronomic crops, though nutrient availability and pH are less well understood in loblolly pine. Besides nutrient availability, nutrient limitations may also arise from lowering the cation exchange capacity (CEC) of the soil in clay mineralogies with pH-dependent charge or by decreasing soil microbial activity (Dai et al. 1998; Thomson et al. 1996).

Keeping soil pH at optimum levels for a given crop allows soil nutrients to be more readily available for plant consumption (Fernández and Hoeft, 2009). It is not common practice in the southeastern United States to lime loblolly pine stands to increase soil pH. Calcium fertilizer, such as lime, raises the pH of soil because it contains anion carbonate (CO_3^{2-}) (Helyar and Anderson, 1974). However, liming loblolly pine stands is common practice in South America. Rotation, planting to harvest, in the southern US is commonly twenty-five years, where in South America it can be as short as sixteen years to complete a rotation. Many researchers have expressed concerns regarding nutrient depletion in the soil from consecutive loblolly pine plantings and the repercussions this may have to sustainable forest production if nutrients are not replaced (Albaugh et al. 2007, Fox et al. 2007a, Vogel and Jokela 2011). After three successive stand rotations of loblolly pine, calcium (Ca) availability was reduced to 32 inches deep (Gatiboni et al. 2020). Ca content can affect canopy robustness, leaf form, quality of wood and height (Littke and Zabowski 2007).

In this study, we examine the impact of repeated N and P fertilization on soil pH across a variety of soils in the southeastern US. Our main objectives are to: (i) determine if frequency of fertilizer application affects changes in soil pH where cumulative rates are similar, (ii), determine if soil pH is significantly lowered on an incremental basis to a one meter depth as cumulative rates of N increase , (iii) evaluate if soil texture or drainage class influence changes in pH with increasing rates of fertilization, and (iv) determine if growth response of the trees is negatively impacted by decreasing soil pH, and (v) evaluate how changes in soil pH affect base saturation and cation exchange capacity. We hypothesize that the more often a site is fertilized the soil will become more acidic. We hypothesize that coarse textured soil pH will be impacted more than fine textured soil due to lower buffering capacity. We also hypothesize that poorly drained soil

pH will be impacted to a lesser degree than well-drained soils as poorly drained soils are less prone to leaching.

Methods and Materials

Study Sites

The study was established in operational loblolly pine plantations between the years 1998 and 2003 across seven different southeastern states of the United States (Figure 1.1). Ages of the trees at study establishment ranged from two to six years old. Sites were selected to comprise a wide range of different soil textures, from clayey to sandy and different drainage classes, poorly drained to extremely well drained (Table 1.1). Initial stocking of the fifteen stands used in the study fell between 1, 248 to 2,246 trees per hectare.

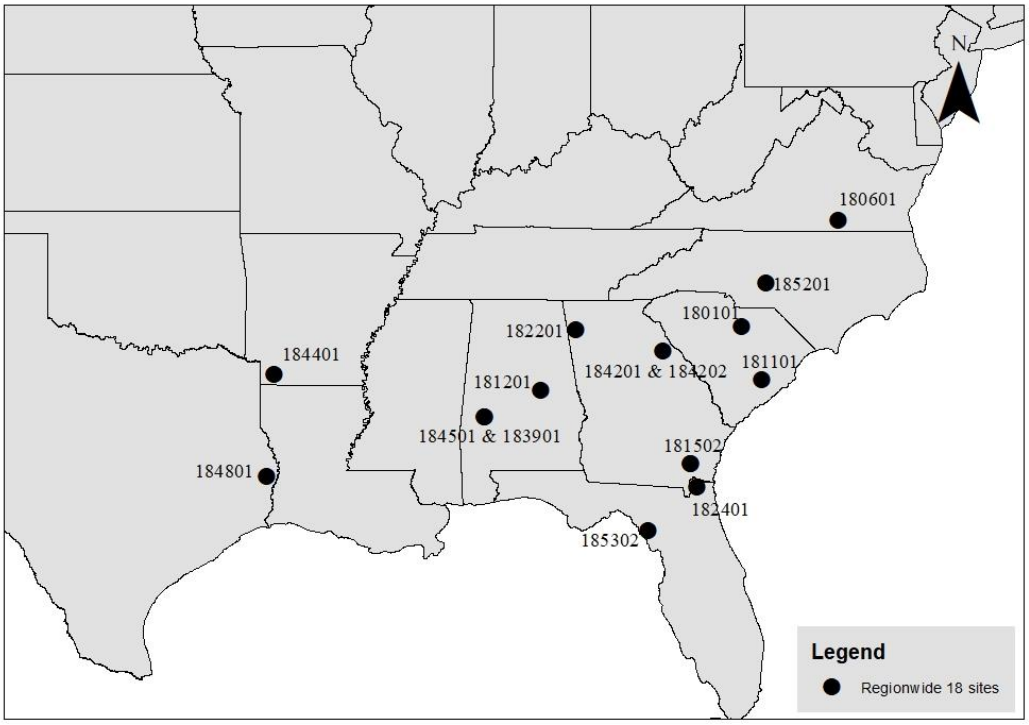


Figure 1.1. Map of study site locations across southeastern US with study site codes.

Table 1.1. Site codes, locations, establishment year, age at establishment, FPC soil code, profile texture, drainage class and treatment replication.

Site Code	Location	Est. Year	Age at Est	FPC Soil Code	Profile Texture	Drainage Class	Treatment Replication
1502	Floyd, GA	2001	2	A	Clayey	Well	2
4501	Marengo, AL	2000	4	A	Clayey	Well	2
2401	Nassau, FL	1999	5	A	Clayey	Poorly	2
601	Brunswick, VA	1999	6	B	Fine Loamy	Well	4
2201	Wilkes, GA	2000	3	B	Fine Loamy	Well	2
1201	Coosa, AL	2002	6	B	Fine Loamy	Well	2
1101	Berkeley, SC	1999	5	B	Fine Loamy	Poorly	4
4801	Newton, TX	2000	3	B	Fine Loamy	Poorly	2
3901	Marengo, AL	2001	3	C	Coarse Loamy	Moderately Well	2
4401	Bradley, AR	2001	3	C	Coarse Loamy	Well	2
4201	Brantley, GA	1998	3	D	Spodic	Poorly	2
4202	Brantley, GA	1998	2	D	Spodic	Poorly	2
5201	Montgomery, AL	2003	4	E	Silty	Extremely Well	2
5302	Dixie, FL	2003	3	F	Deep Subsoil	Poorly	2
101	Kershaw, SC	2000	4	G	Sandy	Well	2

Table 1.2. Study sites grouped by texture class and drainage class.

Soil Drainage and Texture	Clayey	Coarse Loamy	Silty	Sandy
Poorly	2401 (FL) 1101 (SC) 4801 (TX)	4201 (GA) 4202 (GA) 5302 (FL)	—	—
Well	601 (VA) 1502 (GA) 4501 (AL) 2201 (GA) 1201 (AL)	3901 (AL) 4401 (AR)	5201 (NC)	101 (SC)

Experimental Design

The experimental design used in this study was a randomized complete block design. The sites carried two or four replications of each treatment. Plot sizes varied with stand density but had to include sixty measurement trees covering approximately 0.045 hectares. Measurement plots were either 4 rows x 15 trees/row or 6 rows x 10 trees/row. The measurement plot was enclosed within a treatment plot. Most plot dimensions included a treatment plot that measured 35 x 35 meters with an internal measurement plot of 20 x 20 meters.

Nitrogen (N) and Phosphorus (P) were added as urea (46% elemental nitrogen) and diammonium phosphate (18% elemental N, 20% elemental P). Each treatment received phosphorus at a rate of 10% of the N rate. The amount of N in the diammonium phosphate was included in cumulative amount of N applied.

This study included six different treatments, one control and five fertilized treatments (Table 3). The control treatment received 0 kg ha^{-1} N and 0 kg ha^{-1} of P over the course of twelve years. Treatments were fertilized once every two years at 66 or 200 kg ha^{-1} or four years at rates of 133, 200, or 266 kg ha^{-1} N for final cumulative applications of 0, 400 (applied every two or four years), 600, 800, and 1200 kg ha^{-1} N. Material was broadcast by hand or with a hand crank

seeder over each plot. Operational vegetation control was used across all plots with chemical appropriate for the vegetation at each site.

Table 1.3. Amount of nitrogen fertilizer applied over twelve years, frequency of treatment, and amount applied per fertilization.

Total N Applied over 12 years (kg ha^{-1} N)	Treatment Frequency (Years)	Amount Applied Each Fertilization (kg ha^{-1} N)
0	0	0
400	2	66
1200	2	200
400	4	133
600	4	200
800	4	266

Volume Growth Measurement

Volume growth was calculated at plot level. Tree height and diameter at breast height (DBH) measurements were taken for every tree in the measurement plots. Tree height was measured with a hypsometer and DBH was measured at 1.3 meters off the ground with a DBH tape. Volumes were calculated on a per tree basis (Tasissa et al. 1997). The volume of each tree was then summed to get total volume growth of the plot and then converted to a per ha basis. Total volume growth was then divided by 12 years to determine annual average volume growth.

Soil Sampling

Soil samples down to one meter in depth were collected twelve years after study initiation. Surface mineral soil was sampled randomly at five different locations in the measurement plot with a soil auger (6 cm diameter). These samples were broken down into two depth increments: 0-15 cm and 15-30 cm. Additionally a sixth random location within the measurement plot was selected and with the same soil auger, a soil sample was taken from each of the following depths: 0-15 cm, 15-30 cm, 30-50 cm, 50-75 cm, and 75-100 cm.

The mineral soil samples were air-dried at room temperature for one month. Dried samples were then ground using a mortar and pestle. Ground soil was then passed through a 0.02 mm sieve; coarse fragments larger than 0.02 mm were rejected and not used in the soil analysis.

Physical and Chemical Analysis

Using the hydrometer method, particle size (% clay, silt and clay) was determined and then soil texture class was assigned (Ashworth et al. 2001). Soil pH was determined by the Virginia Tech Soil Testing Lab for 13 of the 15 sites. Two of the study sites soil pH was determined in the NC State Forest Productivity Cooperative Soil Lab. All soil pH was determined using a combination glass electrode in a 1:1 soil/water by volume solution (Thomas 1996).

Statistics

A mixed model was used to examine the fixed effects in this study including: frequency of treatment, amount of N + P fertilizer applied for groups of soil texture and drainage classes, respectively, by soil depth increment. Data from different sampling depths were not analyzed against one another to find statistical differences between depth groups. Study site was treated as a random effect. Soil pH differences among treatments were tested using the Tukey's separation of means at an alpha level of 0.05. Linear, quadratic, and exponential regressions models were run for volume versus 0-15 cm soil pH, and base saturation versus soil pH. Regressions with the best R^2 values were selected. Base saturation versus soil pH analysis was modeled at a soil texture group level, with all depths analyzed together. Exponential regressions were modeled on Clayey, Coarse Loamy, and Sandy soil texture groups, and linear regression on Silty texture group. All statistics were performed with JMP (JMP, Version 15. SAS Institute Inc., Cary, NC, 1989-2021).

Results

Frequency of treatment

Two treatments of the study ended the twelve years with both receiving a total of 400 kg ha^{-1} N to test for the effect of frequency (Table 1.3). The effect of frequency of application (every two years or four years) was not statistically significant (p = 0.977; Figure 1.2). The two treatments were statistically similar in every soil group and every individual study site. Frequency of treatment plays no role in lowering soil pH in conjunction with nitrogen fertilizer. For the rest of the statistical analysis the two 400 kg ha^{-1} N treatments are lumped together as one treatment.

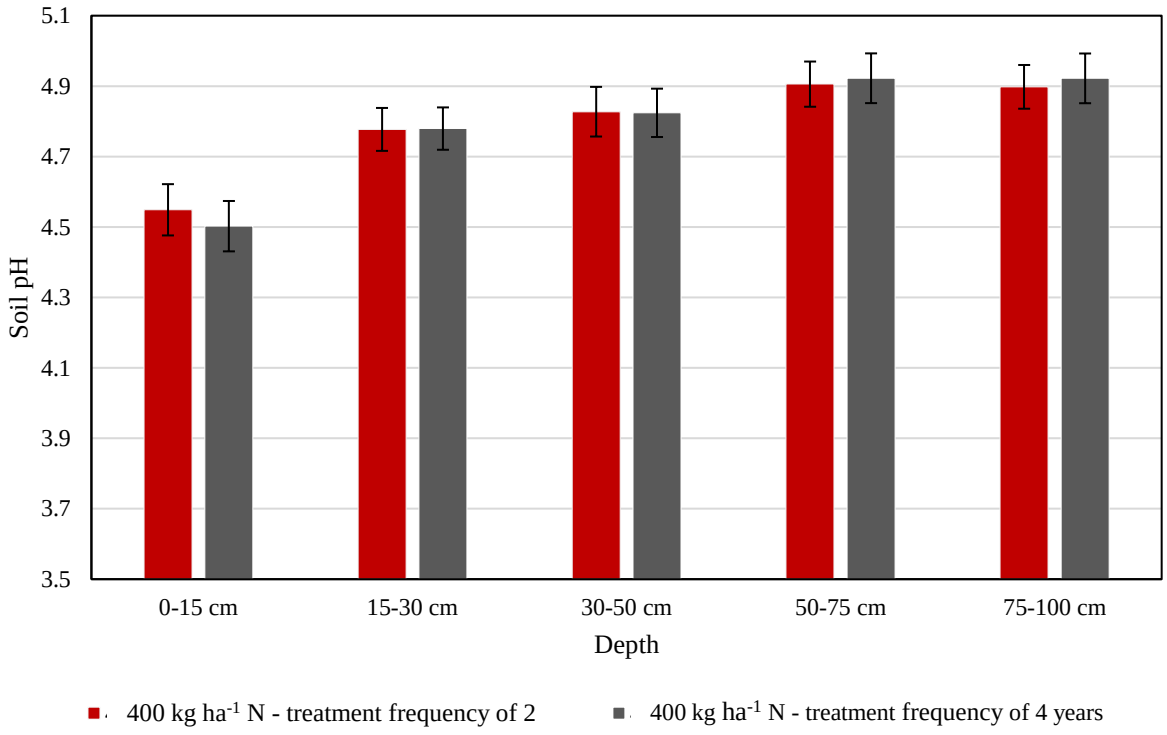


Figure 1.2. Both 400 kg ha^{-1} N treatments mean soil pH from all sites at each soil sampling depth. Red bars represent treatment frequency of every two years and gray bars represent treatment frequency of every four years. Error bars represent stand error of the model. No significant differences were found at a p = 0.05 significance level

All Site Comparison

When all the study sites were analyzed together, the results were statistically significant at four of the five depths. At depth 0-15 cm, all the treatments were significantly lower than the control ($p < 0.0001$). At soil depths 15-30 cm, treatments 600 kg ha^{-1} N, 800 kg ha^{-1} N and 1200 kg ha^{-1} N soil pH was significantly lower than the control ($p < 0.0001$). At depths 30-50 cm ($p < 0.0001$), 50-75 cm ($p = 0.0013$) and 75-100 cm ($p = 0.0001$) treatment 1200 kg ha^{-1} N soil pH was more acidic than the control (Figure 1.3).

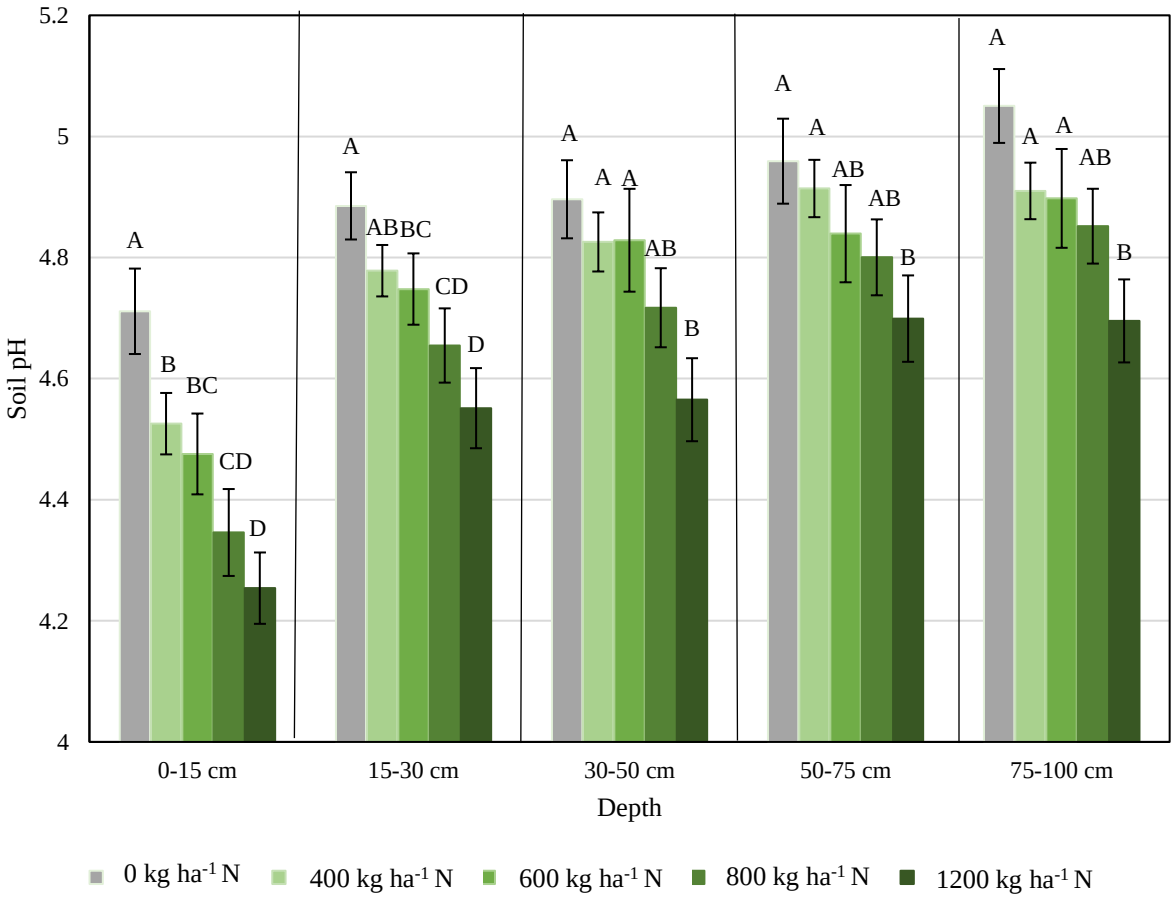


Figure 1.3. Soil pH of all treatments for all sites together at depths were collected at. Error bars represent standard error of the model. Treatments that have similar letter are not significantly different at $p = 0.05$ significance level.

Soil Texture

Soil texture group clayey showed significance decreases in soil pH due to increasing nitrogen rate at all profile increments down to one meter (Figure 1.4). Soil pH was lower in all the treatments than the control at 0-15 cm ($p < 0.0001$). At 15-30 cm ($p < 0.0001$) soil depth, 800 (pH = 4.76) and 1200 kg ha^{-1} N treatments (pH = 4.64) were lower than the control (pH = 5.06). At 30-50cm ($p <0.0001$) and 50-75 cm ($p = 0.0002$) soil depth, 800 and 1200 kg ha^{-1} N treatments had soil pH lower than the control. The 1200 kg ha^{-1} N treatment (pH = 4.78) had a lower pH than the control (pH = 5.18) at 75-100 cm ($p = 0.0009$) while other treatments fell in between.

Soil texture group Coarse Loamy group soil pH was not impacted by nitrogen fertilization (Figure 1.5). In soil texture group Silty, treatments 400 and 1200 kg ha^{-1} N soil pH was significantly lower than from the control at depth 0-15 cm ($p = 0.0144$). Treatment 1200 kg ha^{-1} N soil pH, at depth 15-30 cm, is different from not just the control, but all of the other treatments as well ($p = 0.0026$) (Figure 1.6). Soil texture group Sandy, at depth 0-15 cm, showed that the higher N rates 800 and 1200 kg ha^{-1} N soil pH were significantly lowered compared to the control ($p = 0.0222$). At soil depth 30-50 cm, the two largest treatments soil pHs were different from one another as treatment 800 kg ha^{-1} N soil pH (pH =5.23) was higher than treatment 1200 kg ha^{-1} N soil pH (pH = 4.77) , but comparable to the remaining treatments ($p = 0.0451$; Figure 1.7).

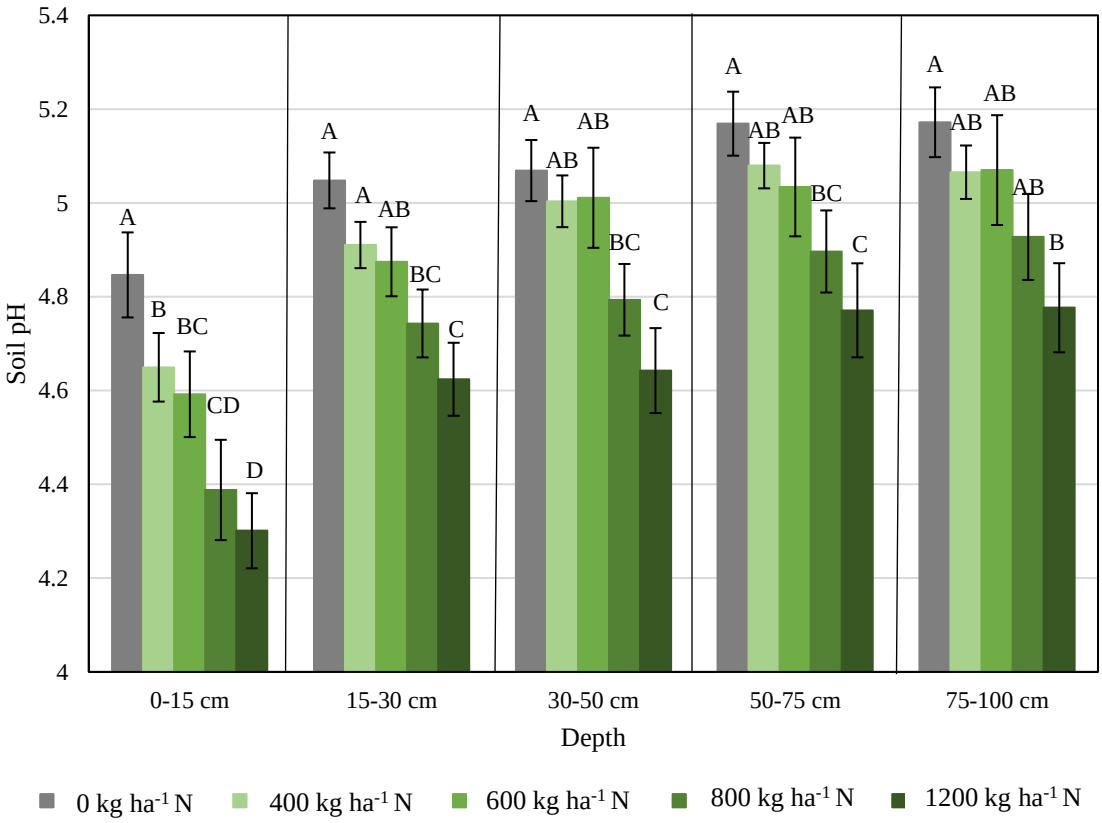


Figure 1.4. Soil texture group Clayey mean soil pH of treatments by depth soil sample was collected. Eight sites make up clayey texture group. Error bars represent standard error of the model. Treatments that have similar letter are not significantly different at p = 0.05 significance level.

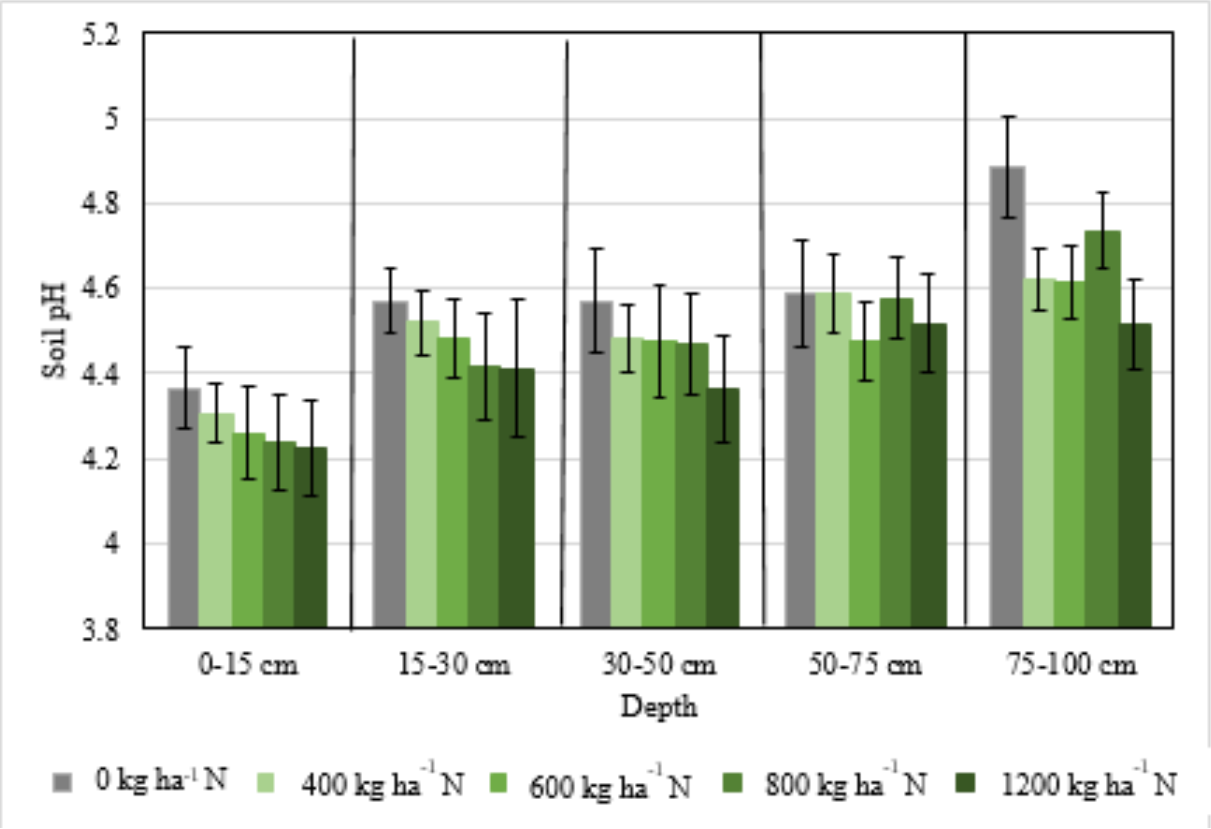


Figure 1.5. Soil texture group Coarse Loamy mean soil pH of treatments by depth soil sample was collected. Five sites make up coarse loamy texture group. Error bars represent standard error of the model. No significant differences were observed at p = 0.05 significance level.

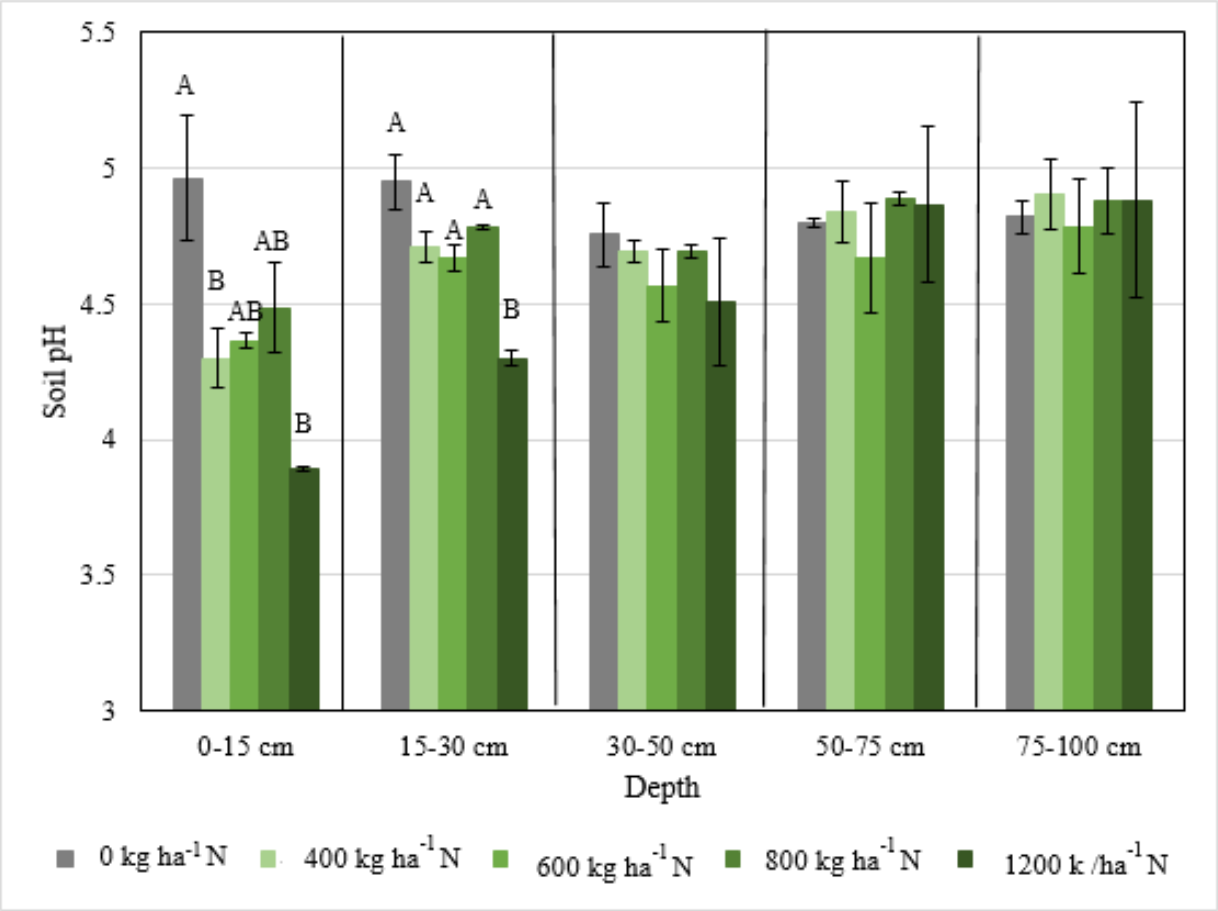


Figure 1.6. Soil texture group Silty mean soil pH of treatments by depth soil sample was collected. One site made up texture group silty. Error bars represent standard error of the model. Treatments that have similar letter, or groups with no letters, are not significantly different at p = 0.05 significance level.

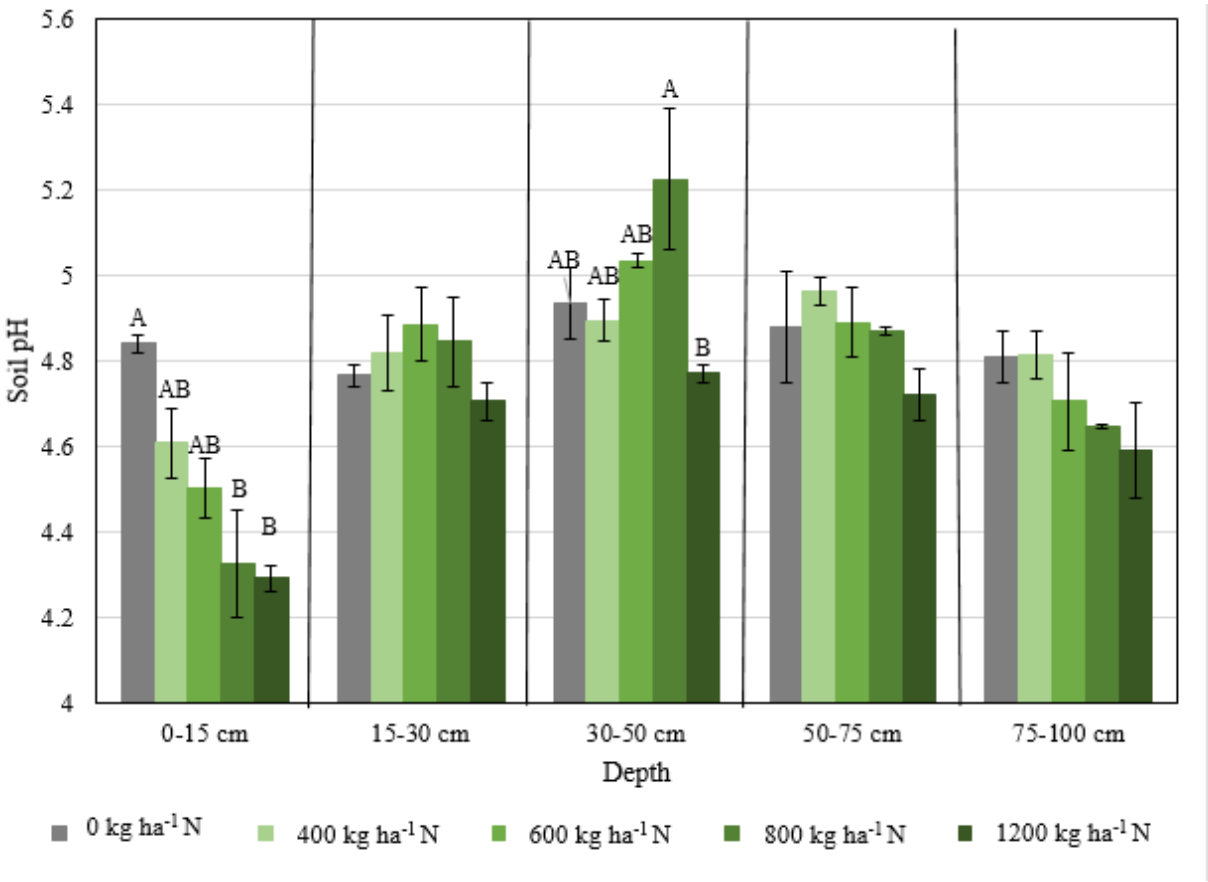


Figure 1.7. Soil texture group Sandy mean soil pH of treatments by depth soil sample was collected. One site made up texture group sandy. Error bars represent standard error of the model. Treatments that have similar letter, or groups with no letters, are not significantly different at p = 0.05 significance level.

Soil Drainage Class

There was a significant interaction between drainage class and nitrogen fertilization in lowering soil pH. Soil pH was affected in more soil depths by nitrogen fertilization in poorly drained sites than well-drained soils (Figure 1.8 and 1.9). In the poorly drained sites at the 0-15 and 15-30 cm depths, all treatments were significantly lowered soil pH compared to the control ($p < 0.0001$) (Figure 1.8). Treatments 800 (pH = 4.45) and 1200 kg ha^{-1} N (pH = 4.3) reported significantly lower soil pH when compared to the control soil pH (pH = 4.73) at depths 30-50 cm ($p = 0.0002$). At depth 50-75cm treatments 800 and 1200 kg ha^{-1} N soil pH were the same at 4.51 and significantly lower than the control at 4.82 ($p = 0.008$). At 75-100 cm soil depth all treatments were significantly lower than the control again ($p = 0.0003$).

Drainage class group well also reported soil pH significantly influenced by nitrogen fertilization, at depths 0-15 cm ($p < 0.0001$), 15-30 cm ($p = 0.0401$), and 30-50 cm ($p = 0.0041$) (Figure 1.9). The 800 (pH = 4.58) and 1200 kg ha^{-1} N (pH = 4.42) treatments soil pH was significantly lower than the control (pH = 4.89). The 800 kg ha^{-1} N (pH = 4.58) treatment soil pH was comparable to 400 (pH = 4.74) and 600 kg ha^{-1} N (pH = 4.7) treatments soil pH at 0-15 cm. At depth 15-30 cm, treatment 1200 kg ha^{-1} N significantly lowered soil pH to where it was not comparable to the control soil pH ($p = 0.0401$). At 30-50 cm soil depth the 1200 kg ha^{-1} N (pH = 4.71) treatment was significantly lower than the control (pH = 4.99) ($p = 0.0041$).

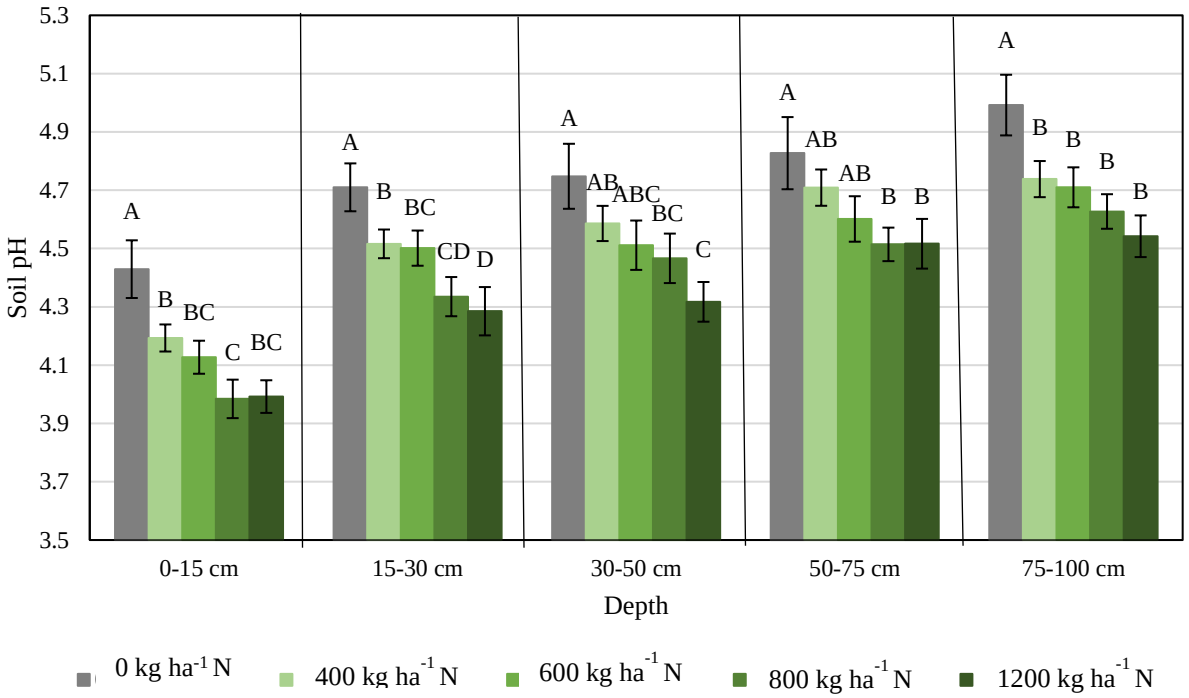


Figure 1.8. Soil drainage group Poorly Drained mean soil pH of treatments by depth soil sample was collected. Error bars represent standard error of the model. Treatments that have similar letter, or groups with no letters, are not significantly different at p = 0.05 significance level.

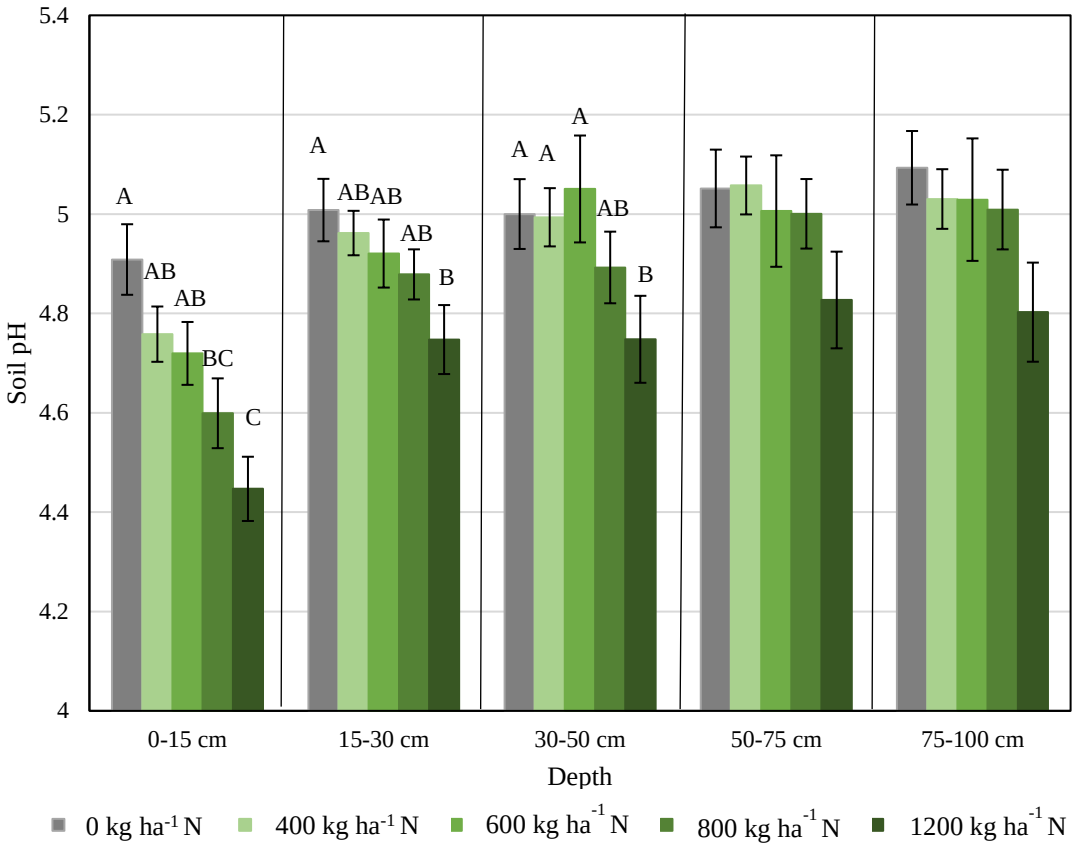


Figure 1.9. Soil drainage group Well Drained mean soil pH of treatments by depth soil sample was collected. Error bars represent standard error of the model. Treatments that have similar letter, or groups with no letters, are not significantly different at p = 0.05 significant.

Base Saturation and Cation Exchange Capacity

Base saturation was significantly lower than the control in treatments 800 kg ha^{-1} N and 1200 kg ha^{-1} N (Figure 1.10) ($p < 0.0001$). Base saturation for the 600 kg ha^{-1} N treatment was similar to the 800 and 1200 kg ha^{-1} N, but not different from the control or 400 kg ha^{-1} N treatment.

Base saturation increased exponentially with increasing soil pH in the soil texture Clayey, Coarse Loamy, and Sandy groups (Figures 1.11, 1.12, and 1.14). Comparison of R^2 values showed exponential relationships fit the data better than linear regressions. Base saturation

increased linearly with increasing soil pH in the soil texture group Silty (Figure 1.13). The R^2 value showed linear relationship matched the best. Cation exchange capacity showed no change with increasing rates of nitrogen fertilization across all study sites or at any depth (Figure 1.15). Mean CEC was greater for texture group clayey than for texture group coarse loamy (Figure 1.16).

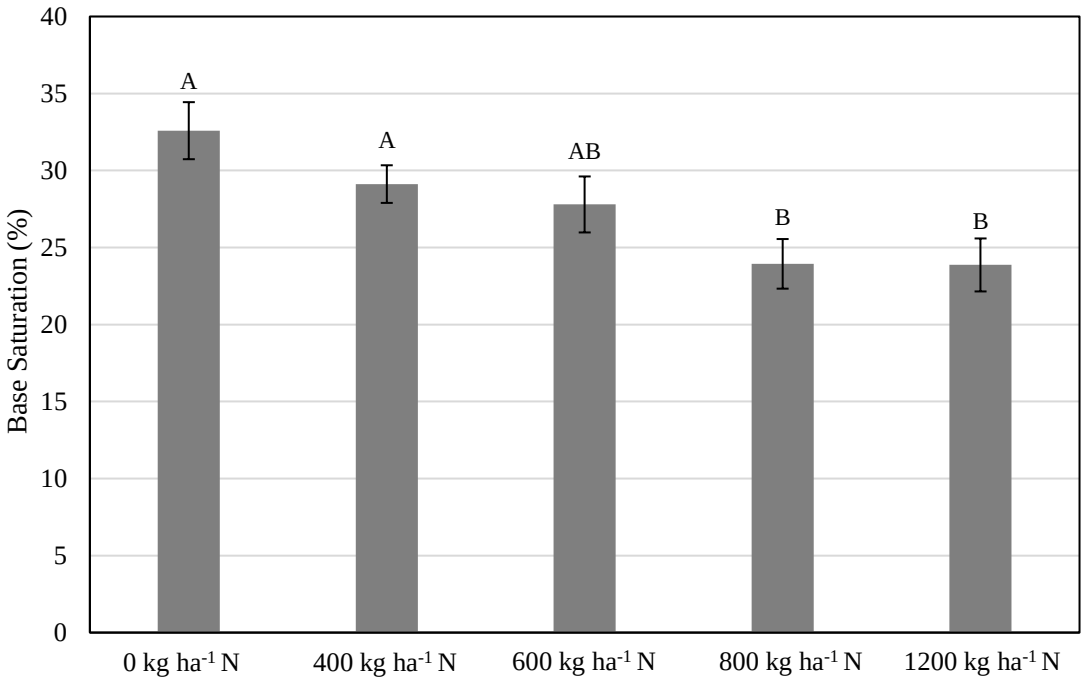


Figure 1.10. Average % base saturation for treatments from all sites and at all depths combined. Letters with similar letters were not significantly different from one another at p = 0.05.

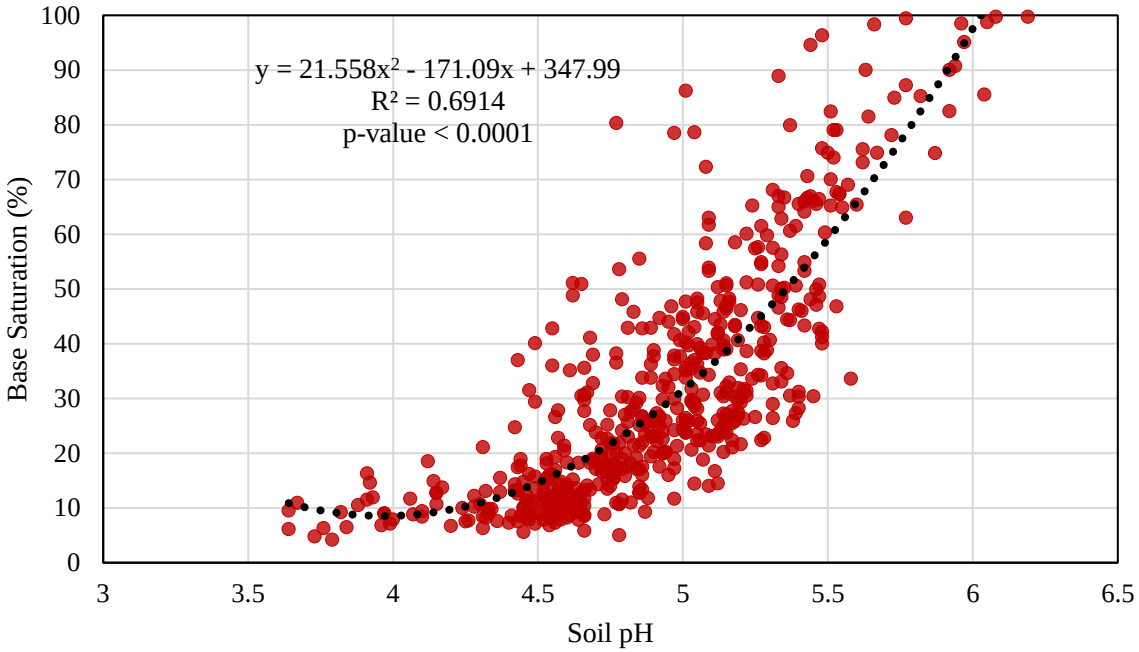


Figure 1.11. Base saturation (%) versus soil pH from texture group Clayey with quadratic formula trend line.

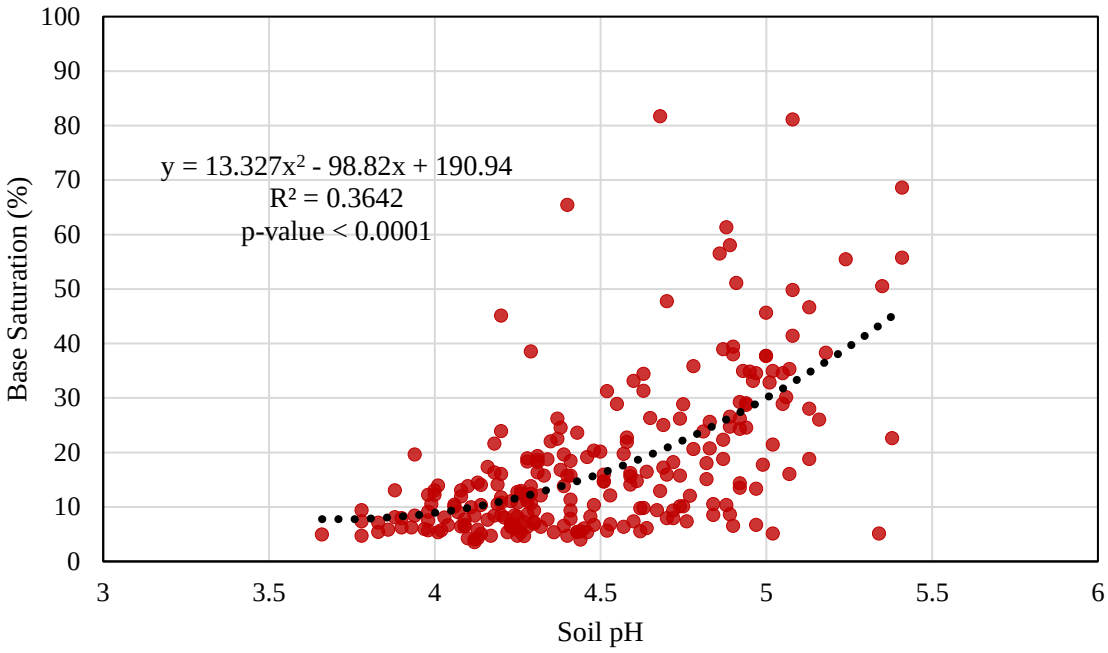


Figure 1.12. Base saturation (%) versus soil pH from texture group Coarse Loamy with quadratic formula trend line.

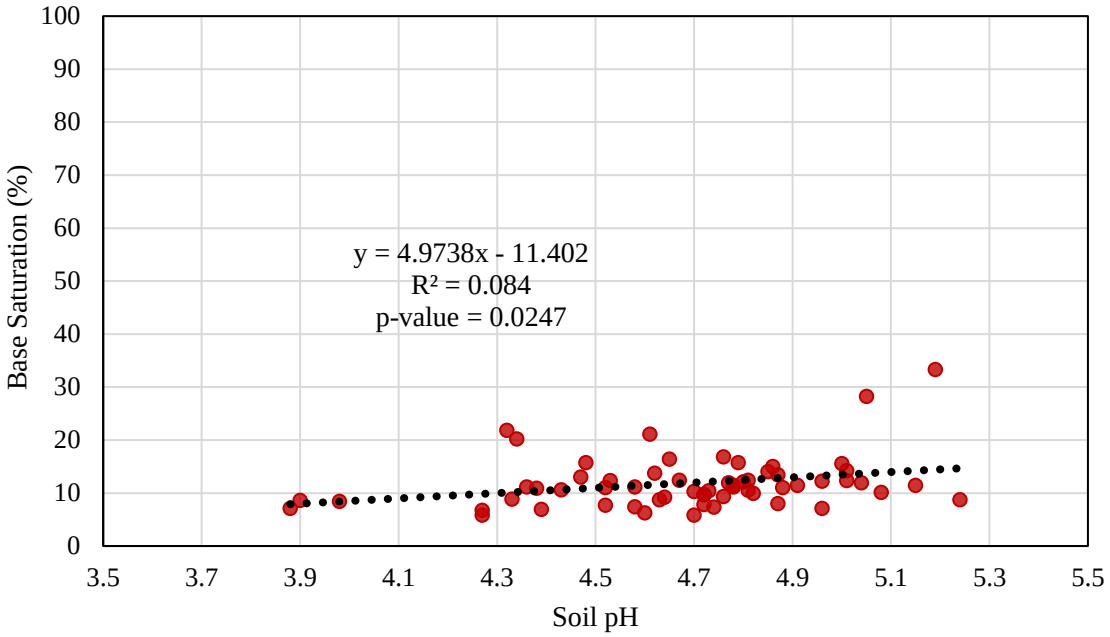


Figure 1.13. Base saturation (%) versus soil pH from texture group Silty with linear formula trend line.

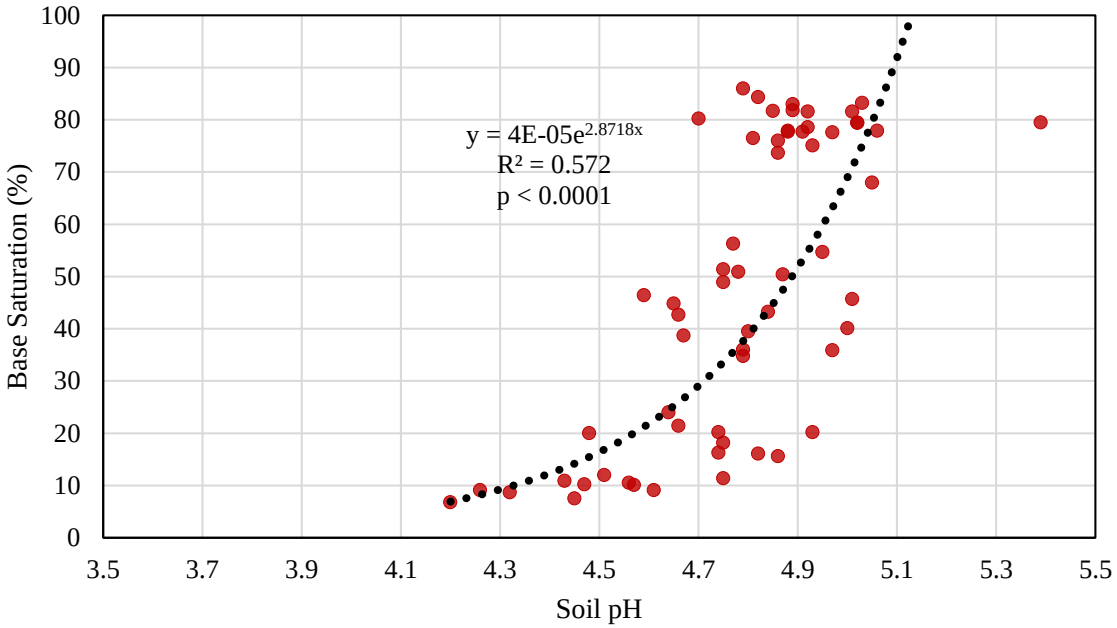


Figure 1.14. Base saturation (%) versus soil pH from texture group Sandy with exponential formula trend line.

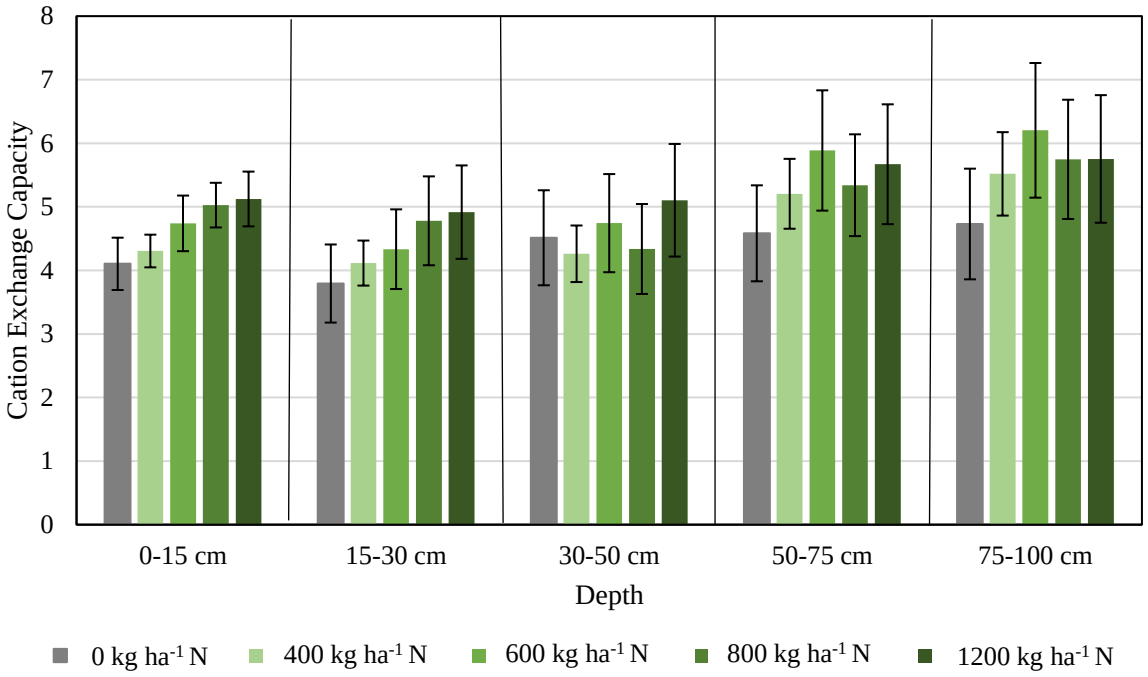


Figure 1.15. Cation exchange capacity of treatments from all sites and all sampling depths. Treatments that have similar letter, or groups with no letters, are not significantly different at p = 0.05 significance level.

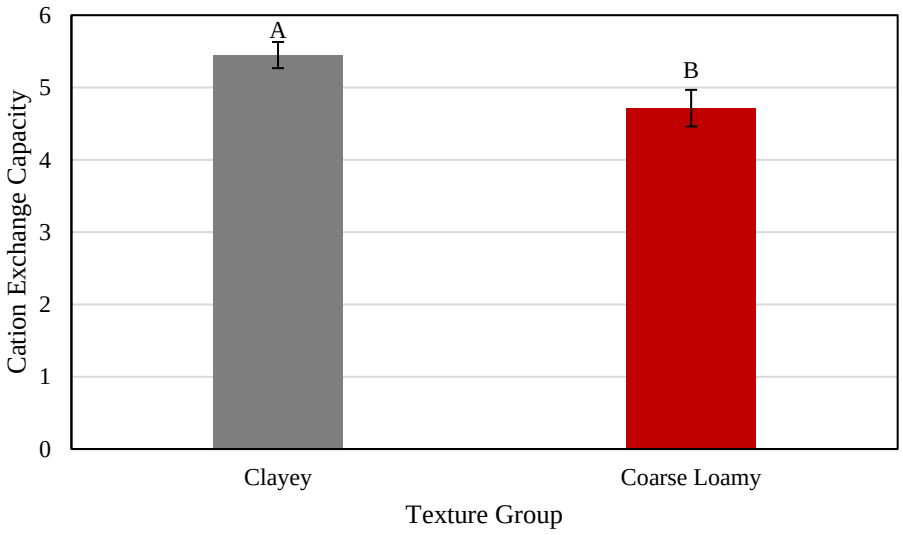


Figure 1.16. Mean cation exchange capacity from all depths of texture groups clayey and coarse loamy. Significantly different from one another at p = 0.0219.

Average annual volume growth vs. soil pH

No relationship was detected between average annual volume growth over the 12-year study period and 0-15 cm soil pH (Figure 1.17).

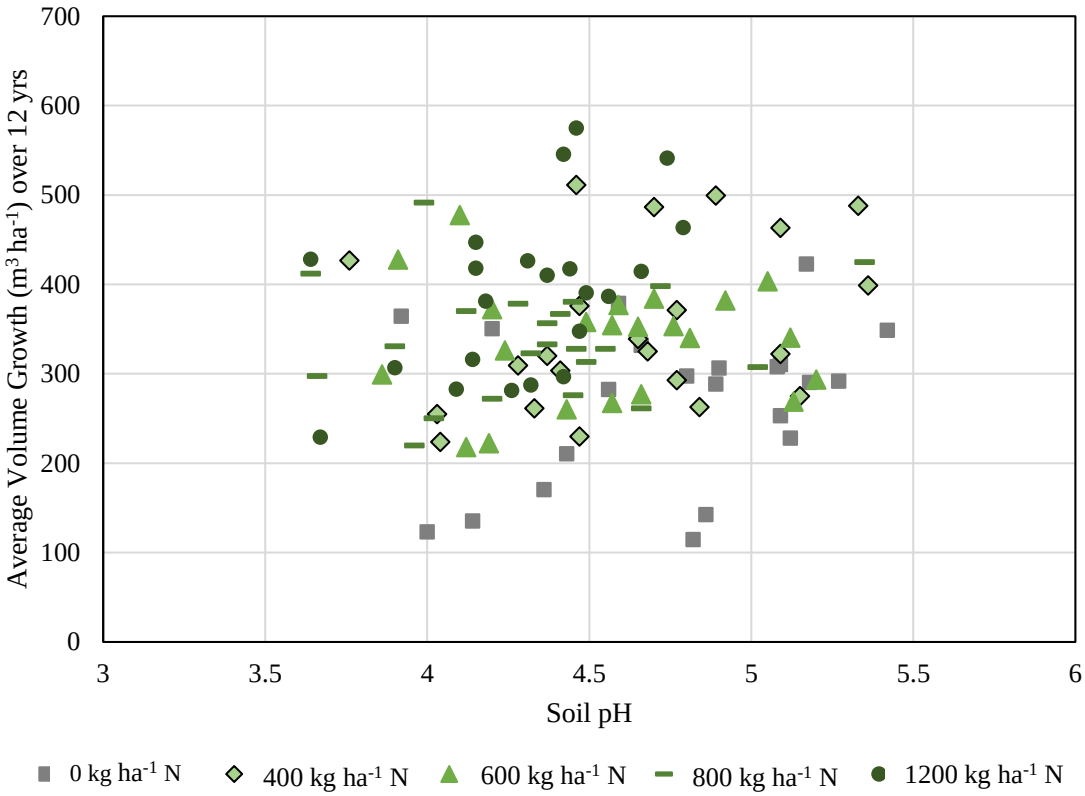


Figure 1.17. Average volume growth (m^3 ha^{-1}) over 12-year study period versus 0-15 cm soil pH from all sites and of all treatments. Differences between treatments not found at a p = 0.05 significance level.

Discussion

Frequency of Treatment

The frequency of treatment, fertilization every two years versus every four years, had no effect on lowering soil pH, which leads us to reject our first hypothesis. This result contrasts with a study on Douglas-fir trees, where urea was applied in three different dosages, annually or every five years. Similar to this study, Tschaplinkshi et al. (1991) found in a sycamore stand that quarterly and annual urea applications did result in lower soil pH than the control, but not from each other (Tschaplinkshi et al. 1991). Only few studies have investigated the effects of different frequencies of N fertilization on forest soils, and conclusions vary on whether fertilizations that are more frequent acidify the soil more than less frequent fertilizations. On the other hand, many of these studies do agree that more frequent fertilization leads to increased leaching (Tschaplinkshi et al. 1991, McNulty and Aber 1993, Johnson and Todd 1988).

All Data Together

When all the data was compiled together, soil pH was affected by the larger treatments down to a meter. This indicates that a portion of the N fertilizer that was not utilized by the plant was leached downward through the soil profile. Bouman et al (1995) found increased N content of the soil from their larger treatment plots over their control plots down to 1.5 meters after nine years of annual fertilization.

Little acidification occurs when the amount of the N fertilizer applied is matched to the N requirement of the plant (Bouman et al. 1995) because when NO_3 is taken up by plants, anions are released to the soil, compensating the acidity generated by the fertilizer. Treatments 400 and 600 kg ha^{-1} N in texture group clayey have soil pH that are similar to the control treatment soil

pH from 30 cm down to a meter, indicating they are good matches for loblolly pine N requirements as acidification only occurred at the very top of the soil profile. Treatment 800 kg ha^{-1} N did acidify the soil noticeably from the control in the top layers of the soil profile, which would indicate that this treatment is more than loblolly pine N requirements. Treatment 1200 kg ha^{-1} N is excessive, as it was determined that the most efficient amount of N to apply was 400 kg ha^{-1} N (Albaugh et al. 2015), the N not utilized by the plant stayed in the soil and acidified the soil probably past the meter mark.

Soil Texture

Nitrogen fertilizer and soil texture had an effect on lowering soil pH. Our hypothesis was supported that soil pH would be affected by different soil textures, but not supported by which would have soil pH lowered to a greater degree. We expected that more coarse textured soils would be greatly affected by fertilization, instead of finer textured soils. Coarser textured soils are generally less buffered than soils of fine textured soils, except for kaolinite clay soils, which is the primary clay mineral in the southeastern US, where our study is located throughout (Weaver et al. 2004).

Another explanation of why clay soil had soil pH lowered and not coarse loamy is the difference between the two groups CEC. In fine-textured soils, excessive N is converted to nitrate (NO_3), and the resulting H are attracted to the CEC of the soil when the excess NO_3 is leached past the rooting zone. Excessive N is also converted to nitrate in more coarse textured soils but due to a lower CEC, H does not accumulate as much as in finer textured soils. Coarser textured soils, due to lower CEC as well, are more prone to increased ammonia (NH_3) loss (Ohnemus et al. 2021). Nitrogen fertilization rate recommendations to achieve optimal growth of

annual crops takes into consideration the soil texture of the field. If the field is of a coarse texture than finer texture, a higher N rate is recommended to compensate for N that will be leached out or lost to the conversion of NH_3 (Alotaibi et al. 2018 and Cambouris et al. 2016). Hansen and Djurhuus (1996) found that more leaching accord on coarser soils when there was less vegetation, a "catch crop", was not present. As all of our plots had operational weed control, i.e. the understory was present but not overrunning the trees; it would not be surprising if this led to a decrease in leaching past the rooting zone in the coarse loamy group.

Our clayey soil texture group had significant results down to one meter. For soil fertility tests, the recommendation is to soil sample just to 7 cm (Reeves and Liebig 2016). Acidity is most profound in the top few centimeters, this held true for our study as well with all 0-15 cm soil pH being the lowest. Management decisions for fertilization should be based off the top few centimeters of soil, because that is where plants intercept most of their nutrients (Reeves and Liebig 2016). Miller et al (2006) found that loblolly pine stands that had more competition tended to have more coarse-root biomass in the top 30 cm of soil, while trees that had very little competition had an even vertical distribution of roots, but in both situations 90% of roots occupied the top 50 cm of soil. On the other hand, after soil has been through intensive management, i.e. multiple fertilizations and logging, it is important to know how the soil has been altered (Slesak 2013). Most studies only soil sample to 30 cm or so, if that had been done in this study we would never have known the impact treatment 1200 kg ha^{-1} N had on soil pH of clayey soils. Effects of that treatment are still felt down to a meter, which strengthens the argument Slesak (2013) makes for more intensive soil sampling post-intensive management.

Though coarser textured soils require more N fertilizer to achieve optimal growth than clayey soils (Alotaibi et al. 2018 and Cambouris et al. 2016), they require less liming to readjust

soil pH. Clayey soils require more lime fertilizer to raise pH, due to higher CEC (Delin 2005). Finer textured soils have an inherent ability to hold on to nutrients and additives much more than coarser soils. It is therefore logical to think that finer textured soils would "reap" the benefits or repercussions of a treatment more than a coarser soil.

Soil Drainage

Our hypothesis that poorly drained soil pH would be impacted to a lesser degree by N fertilization than well drained soils was rejected. In general, poorly drained soils had pH lowered to a greater depth than more well drained sites of similar textures. Poorly drained soils are less prone to leaching, which means the N fertilizer is in the soil system longer and has more time to acidify the soil (Fernandez et al. 2017). Nitrate moves through soil at the same velocity as water, if not taken up by plants (Kookana and White 1998). If the water is not moving from the site and there is little oxygen in the soil the process of denitrification can occur, converting NO_3 to a gas lost to the atmosphere (Hofstra and Bouwman 2005).

The first step in the Fuzzy Theory, decision support system for N fertilization, is determining the mineralization potential of a site (Papadopoulos et al. 2011). To determine mineralization potential the sites soil pH, clay content, soil temperature and drainage class are evaluated. Soil drainage is closely linked to N mineralization, but does increased mineralization lead to increased plant uptake? In two soils of the same texture, sandy loam, Kopp and Guillard (2002) found that the poorly drained site had better N mineralization than the well-drained site, but found no additional plant uptake at the poorly drained site over the well-drained site. Fernandez et al. (2017) found no growth differences between fertilized drained and undrained sites. On the other hand, another study found that during dry years poorly drained sites had better

plant uptake than well drained sites, but during wet years both sites had similar plant uptake (Welegedara et al. 2020).

Base Saturation and CEC

Base saturation (BS) and cation exchange capacity (CEC) are closely tied and dependent on soil pH. As soil pH decreases, one would expect BS and CEC to decline as well. In this study, BS diminished as soil pH levels fell. In a study on the forest floor of Douglas fir, repeated urea applications did not result in diminished soil pH or BS in the O horizon of the fertilized plots compared to the unfertilized plots (Prietzel et al. 2004).

In our results, we see that CEC among the different treatments does not vary enough to be deemed significantly different from one another. We anticipated the CEC to decline with the greater additions of N. Another study found that soil pH and base saturation was decreased more from the control as urea fertilizer rates increased, but saw the CEC increase more as fertilization rate grew, most likely due to increased soil organic matter (Cakmak et al. 2010). Though no differences could be found between treatments at any depths, it does appear that CEC of the soil is trending upwards with increasing rates of N in this study. This is likely due to increased soil organic matter as N increases LAI of loblolly pine (Albaugh et al. 1998). Fox et al. (2007b) found CEC was lowered in the annual application treatments but not when fertilization was spaced out over the years. Since trees are not fertilized annually, just two or three times over the entire rotation, lowering CEC does not appear to be a great risk in fertilization of loblolly pine stands. Instead it appears that CEC may increase with N fertilization, due to increased foliage production, which leads to more soil organic matter which increases CEC of the soil.

Volume Growth and Soil pH

Even with the soil pH lowered significantly with increasing rates of fertilization, the average annual growth rate was not negatively impacted during the duration of this study. Growth of the trees was increased with the addition of N up to 400 kg ha^{-1} N (RW18 study paper), but lowering soil pH did not have an adverse effect on the growth of the trees. This result is not surprising, since loblolly pine is known for growing well on acidic soil (4.5 – 6) (Schultz 1997). The majority of commercially grown tree species live in an array of soil pH levels as long as there is a proper balance of essential nutrients (N and P) available (Londo et al. 2006).

This indicates that there is not a link between loblolly growth and a required specific soil pH level for growth as there is for annual rotation crops. N applications are necessary for almost all agronomic, non-nitrogen fixing, crops to sustain productivity (Fageria and Baligar 2005) and have been well connected to decreasing soil pH (Rifai et al. 2010). Lime applications have been used for decades to maintain soil pH at optimum levels, and when used to balance N fertilizer applications have shown no evidence of declining productivity from long-term N fertilizer use on agronomic crops (Bundy et al. 2011).

Only a handful of people have done liming studies concerning loblolly pine in the US. In a study done at the Hofmann Forest on the coast of North Carolina in the 1970s, it was found that a treatment of limestone and P fertilization increased height growth. The soil pH of this treatment raised to 4.1 from 3.5 soil pH of the control; the highest yield was achieved by the lime and P treatment (MacCarthy and Davey 1976). On the coastal plain of Virginia a second study with N included in the lime and P treatment showed that the P + N + lime treatment greatly out preformed the control, P, and P +N treatments in height, DBH, and total volume. Stumpage

value for the P + N + lime treatment was $4,896/ac whereas the other treatments stumpage values ranged from $3,960/ac to $4,045/ac (Kyle et al. 2005).

Limitations and future directions

The biggest limitation of this study was the imbalance of soil texture groups and drainage classes among the study sites. Others soil sampled multiple times throughout their study's time span, recorded rainfall and soil temperature. In an ideal scenario, soil samples would have been collected one month, six months and then every year after fertilization, along with addition of more sites. Collecting rainfall data, along with soil temperature data would have allowed for more in depth analysis of the current data. N transformations in the soil are heavily influenced by moisture and temperature (Fernandez et al. 2017).

Having more study sites that were silty and sandy in texture would help make results from the two we did have more clear and reliable. Flint et al. (2008) found urea fertilization on Douglas-fir, did lower the soil pH of very coarse gravelly soil down to a meter and found that 2% of applied fertilizer was leached down past 100 cm in the soil after one year (Flint et al. 2008). At the sandy group site in this study, soil pH was lowered in the first measurement of 0-15 cm; if more data had been available maybe, our results would be similar to those of Flint.

Five years after N fertilization, no differences detected between fertilized versus non-fertilized plots on total or available N contents of the soil in a grand fir stand (Tiedemann et al. 1998). A study conducted fifteen years post N fertilization of Scots pine and Norway spruce stands found that the N + lime fertilized plots had a higher pH than that of the control (Saarsalmi et al. 2009). It is unclear if soil pH will be restored naturally to pre-fertilization levels years after

N fertilization took place, or if lime additions are the only way to counteract the acidification of the soil.

Future studies will include a P carryover project of these sampled plots so soil pH can be reanalyzed to determine more longer-termed effects of N fertilization on soil pH and if there are any negative growth effects associated with acidification of the soil.

Conclusion

No interaction was detected between frequency of treatment and nitrogen fertilization in lowering soil pH, and other studies found more leaching when treatment frequency is increased. A significant interaction between texture and nitrogen fertilization in lowering soil pH, where finer textured soils pH was lowered from the control by the larger treatments down to a meter. Coarser textured soil pH was unaltered by increasing urea fertilization treatments. A relationship between drainage class and nitrogen fertilizer in lowering soil pH also exists, well and poorly drained soils experienced soil acidification down to 30 cm by the larger treatments. Additionally, the poorly drained soils experienced soil acidification down to a meter. As soil pH lowered, so did base saturation, but CEC remained unaltered. Lowering soil pH did not have an effect on curbing average volume growth. Even though average volume growth was not impacted this growth rotation, the lowering of base saturation may indicate growth problems for future rotations.

REFERENCES

Adams, F. “Crop Response to Lime in the Southern United States.” *Agronomy Monographs* 12 (1984): 211–65. https://doi.org/10.2134/agronmonogr12.2ed.c5.

Albaugh, T, T Fox, H Allen, and R Rubilar. “Juvenile Southern PINE Response to Fertilization Is Influenced by Soil Drainage and Texture.” *Forests* 6, no. 12 (2015): 2799–2819. https://doi.org/10.3390/f6082799.

Albaugh, Timothy J, H L Allen, and T R Fox. “Historical Patterns of Forest Fertilization in the Southeastern United States from 1969 to 2004.” *Southern Journal of Applied Forestry* 31, no. 3 (August 2007): 129–37. https://doi.org/10.1093/sjaf/31.3.129.

Albaugh, Timothy J, H L Allen, P M Dougherty, and L W Kress. “Leaf Area and Above- and Belowground Growth Responses of Loblolly Pine to Nutrient and Water Additions.” *Forest Science* 44, no. 2 (May 1998): 317–28.

Albaugh, Timothy J, T R Fox, R L Cook, and J E Raymond. “Forest Fertilizer Applications in the Southeastern United States from 1969 to 2016.” *Forest Science* 65, no. 3 (June 2019). https://doi.org/10.1093/forsci/fxy058.

Allen, H L, T R Fox, and R G Campbell. “What Is Ahead for Intensive Pine Plantation Silviculture in the South? .” *Southern Journal of Applied Forestry* 29, no. 2 (April 2005): 62–69. https://doi.org/10.1093/sjaf/29.2.62.

Alotaibi, K D, A N Cambouris, M St. Luce, N Ziadi, and N Tremblay. “Economic Optimum Nitrogen Fertilizer Rate and Residual Soil Nitrate as Influenced by Soil Texture in Corn Production.” *Agronomy Journal* 110, no. 6 (2018): 2233–42. https://doi.org/10.2134/agronj2017.10.0583.

Ashworth, J, D Keyes, R Kirk, and R Lessard. “Standard Procedure in the Hydrometer Method for Particle Size Analysis.” *Communication's in Soil Science and Plant Analysis* 32 (2001): 633–42.

Barak, P, B O Jobe, A R Krueger, L A Peterson, and D A Laird. “Effects of Long-Term Soil Acidification Due to Nitrogen Fertilizer Inputs in Wisconsin.” *Plant and Soil* 197 (August 28, 1997): 61–69.

Bouman, O T, D Curtin, C A Campbell, V O Biederbeck, and H Ukrainetz. “Soil Acidification from Long-Term Use of Anhydrous Ammonia and Urea.” *Soil Science Society of America Journal* 59, no. 5 (1995): 1488–94. https://doi.org/10.2136/sssaj1995.03615995005900050039x.

Bundy, L G, T W Andraski, M D Ruark, and A E Peterson. “Long-Term Continuous Corn and Nitrogen Fertilizer Effects on Productivity and Soil Properties.” *Soil Fertility & Crop*

Nutrition 103, no. 5 (September 2011): 1346–51. https://doi.org/https://doi-org.prox.lib.ncsu.edu/10.2134/agronj2011.0094.

Cakmak, Dragan, Elmira Saljnikov, V. Perovic, D. Jaramaz, and V. Mrvic. "Effect of long-term nitrogen fertilization on main soil chemical properties in Cambisol." In *19 th World congress of soil science, soil solutions for changing world, Brisbane, Australia*. 2010.

Cambouris, A N., N Ziadi, I Perron, K D Alotaibi, M St. Luce, and N Tremblay. "Corn Yield Components Response to Nitrogen Fertilizer as a Function of Soil Texture." *Canadian Journal of Soil Science* 96, no. 4 (2016): 386–99. https://doi.org/10.1139/cjss-2015-0134.

Carlson, C A, T R Fox, H Lee Allen, and T J Albaugh. "Modeling Mid-Rotation Fertilizer Responses Using the Age-Shift Approach." *Forest Ecology and Management* 256, no. 3 (2008): 256–62. https://doi.org/10.1016/j.foreco.2008.04.020.

Carlson, C A, T R Fox, H L Allen, T J Albaugh, R A Rubilar, and J L Stape. "Growth Responses of Loblolly Pine in the Southeast United States to Midrotation Applications of Nitrogen, Phosphorus, Potassium, and Micronutrients." *Forest Science* 60, no. 1 (July 11, 2013): 157–69. https://doi.org/https://doi.org/10.5849/forsci.12-158.

Carlson, C A, T R Fox, S R Colbert, D L Kelting, H L Allen, and T J Albaugh. "Growth and Survival of Pinus Taede in Response to Surface and Subsurface Tillage in the Southeastern United States." *Forest Ecology Management* 234 (2006): 209–17.

Croatan Series. National Cooperative Soil Survey, September 2008. https://soilseries.sc.egov.usda.gov/OSD_Docs/C/CROATAN.html.

Dai, Z, Y Liu, X Wang, and D Zhao. "Changes in PH, CEC and Exchangeable Acidity of Some Forest Soils in Southern China During the Last 32-35 Years." *Water, Air, and Soil Pollution* 108 (1998): 377–90.

Delin, S. "Site-Specific Nitrogen Fertilization Demand in Relation to Plant Available Soil Nitrogen and Water: Potential for Prediction Based on Soil Characteristics." Dissertation, Department of Soil Sciences, Swedish University of Agricultural Sciences, 2005.

Fageria, N K, and V C Baligar. "Enhancing Nitrogen Use Efficiency in Crop Plants." *Advances in Agronomy*, 2005, 97–185. https://doi.org/10.1016/s0065-2113(05)88004-6.

Fernández, F G, K P Fabrizzi, and S L Naeve. "Corn and Soybean's Season-Long in-Situ Nitrogen Mineralization in Drained and Undrained Soils." *Nutrient Cycling in Agroecosystems* 107, no. 1 (November 2017): 33–47. https://doi.org/10.1007/s10705-016-9810-1.

Fernandez, F G, and R G Hoeft. Tech. *Managing Soil PH and Crop Nutrients*. Champaign, IL: Illinois Agronomy Handbook, 2009.

Flint, C M, R B Harrison, B D Strahm, and A B Adams. “Nitrogen Leaching from Douglas-Fir Forests AFTER Urea Fertilization.” *Journal of Environmental Quality* 37, no. 5 (September 2008): 1781–88. https://doi.org/10.2134/jeq2007.0367.

Fox, T R, E J Jokela, and H L Lee. “The Development of Pine Plantation Silviculture in the Southern United States .” *Journal of Forestry* 105, no. 7 (October 2007a): 337–47. https://doi.org/https://doi-org.prox.lib.ncsu.edu/10.1093/jof/105.7.337.

Fox, T R, H L Allen, T J Albaugh, R Rubilar, and C Carlson. “Tree Nutrition and Forest Fertilization of Pine Plantations in the Southern United States.” *Southern Journal of Applied Forestry* 31 (2007b): 5–11.

Gatiboni, L C, W C da Silva, and G L Mumbach. “Use of Exchangeable and Nonexchangeable Forms of Calcium, Magnesium, and Potassium in Soils without Fertilization after Successive Cultivations with Pinus Taeda in Southern Brazil.” *Journal of Soils and Sediments* 20 (February 2020): 665–74. https://doi.org/https://doi-org.prox.lib.ncsu.edu/10.1007/s11368-019-02460-x.

Gough, C M, J R Seiler, and C A Maier. “Short-Term Effects of Fertilization on Loblolly Pine (Pinus Taeda L.) Physiology.” *Plant, Cell & Environment*, May 7, 2004. https://doi.org/https://doi.org/10.1111/j.1365-3040.2004.01193.x.

Hansen, E M, and J Djurhuus. “Nitrate Leaching as Affected by Long-Term N Fertilization on a Coarse Sand.” *Soil Use and Management* 12, no. 4 (1996): 199–204. https://doi.org/10.1111/j.1475-2743.1996.tb00543.x.

Helyar, K R, and A J Anderson. “Effects of Calcium Carbonate on the Availability of Nutrients in an Acid Soil.” *Soil Science Society of America Journal* 38, no. 2 (1974): 341–46. https://doi.org/10.2136/sssaj1974.03615995003800020035x.

Hofstra, N, and A F Bouwman. “Denitrification in Agricultural Soils: SUMMARIZING Published Data and Estimating Global Annual Rates.” *Nutrient Cycling in Agroecosystems* 72, no. 3 (February 24, 2005): 267–78. https://doi.org/10.1007/s10705-005-3109-y.

JMP, Version 15. SAS Institute Inc., Cary, NC, 1989-2021

Johnson, D W, and D E Todd. “Nitrogen Fertilization of Young Yellow Poplar and Loblolly Pine Plantations at Differing Frequencies.” *Soil Science Society of America Journal* 52, no. 5 (1988): 1468–77. https://doi.org/10.2136/sssaj1988.03615995005200050050x.

King, John S, T J Albaugh, M Buford, B R Strain, and P Dougherty. “Below-Ground Carbon Input to Soil Is Controlled by Nutrient Availability and Fine Root Dynamics in Loblolly Pine.” *New Phytologist* 154, no. 2 (April 30, 2002): 389–98. https://doi.org/https://doi.org/10.1046/j.1469-8137.2002.00393.x.

Kookana, R S, and R E White. “Measuring Nutrient and Pesticide Movement in Soils: Benefits for Catchment Management.” *Australian Journal of Experimental Agriculture* 38, no. 7 (1998): 725. https://doi.org/10.1071/ea97154.

Kopp, K L, and K Guillard. “Clipping Management and Nitrogen Fertilization of Turfgrass.” *Crop Science* 42, no. 4 (2002): 1225–31. https://doi.org/10.2135/cropsci2002.1225.

Kyle, K H, L J Andrews, T R Fox, W M Aust, J A Burger, and G H Hansen. “Long-Term Effects of Drainage, Bedding, and Fertilization on Growth or Loblolly Pine (Pinus Taeda L.) in the Coastal Plain of Virginia.” *Southern Journal of Applied Forestry* 29, no. 4 (2005): 205–14.

Littke, K M, and D Zabowski. “Influence of Calcium Fertilization on Douglas-Fir Foliar Nutrition, Soil Nutrient Availability, and Sinuosity in Coastal Washington.” *Forest Ecology and Management* 247 (April 2007): 140–48.

Londo, A J, J D Kushla, and R C Carter. “Soil PH and Tree Species Suitability in the South.” *Southern Regional Extension Forestry*, January 2006, 1–4.

MacCarthy, R, and C B Davey. “Nutritional Problems of Pinus Taeda L. (Loblolly Pine) Growing on Pocosin Soil.” *Soil Science Society* 40 (July 1976): 582–85.

McNulty, S G, and J D Aber. “Effects of Chronic Nitrogen Additions on Nitrogen Cycling in a High-Elevation Spruce–Fir Stand.” *Canadian Journal of Forest Research* 23, no. 7 (1993): 1252–63. https://doi.org/10.1139/x93-160.

Miller, A T, H L Allen, and C A Maier. “Quantifying the Coarse-Root Biomass of Intensively Managed Loblolly Pine Plantations.” *Canadian Journal of Forest Research* 36, no. 1 (2006): 12–22. https://doi.org/10.1139/x05-229.

Ohnemus, T, O Spott, and E Thiel. “Spatial Distribution of Urea Induced Ammonia Loss Potentials of German Cropland Soils.” *Geoderma* 394 (July 15, 2021): 1–13. https://doi.org/https://doi.org/10.1016/j.geoderma.2021.115025.

Papadopoulos, A, D Kalivas, and T Hatzichristos. “Decision Support System for Nitrogen Fertilization Using Fuzzy Theory.” *Computers and Electronics in Agriculture* 78, no. 2 (June 2011): 130–39. https://doi.org/10.1016/j.compag.2011.06.007.

Prietzel, J, G L Wagoner, and R B Harrison. “Long-Term Effects of Repeated Urea Fertilization in Douglas-Fir Stands on Forest Floor NITROGEN Pools and Nitrogen Mineralization.” *Forest Ecology and Management* 193, no. 3 (February 1, 2004): 413–26. https://doi.org/10.1016/j.foreco.2004.02.006.

Reeves, Justin L., and Mark A. Liebig. “Depth Matters: Soil PH and Dilution Effects in the Northern Great Plains.” *Soil Science Society of America Journal* 80, no. 5 (2016): 1424–27. https://doi.org/10.2136/sssaj2016.02.0036n.

Rifai, S W, D Markewitz, and B Borders. “Twenty Years of Intensive Fertilization and Competing Vegetation Suppression in Loblolly Pine Plantations: Impacts on Soil C, N, and Microbial Biomass.” *Soil Biology and Biochemistry* 42, no. 5 (2010): 713–23. https://doi.org/10.1016/j.soilbio.2010.01.004.

Saarsalmi, A, A Smolander, M Kukkola, and M Arola. “Effect of Wood Ash and Nitrogen Fertilization on Soil Chemical Properties, Soil Microbial Processes, and Stand Growth in Two Coniferous Stands in Finland.” *Plant and Soil* 331, no. 1-2 (December 18, 2009): 329–40. https://doi.org/10.1007/s11104-009-0256-y.

Schultz, R P. *Loblolly Pine: the Ecology and Culture of Loblolly Pine (Pinus Taeda L.)*. Washington, DC: USDA Forest Service, 1997.

Slesak, R A. “Soil Temperature Following Logging-Debris Manipulation and Aspen Regrowth in Minnesota: Implications for Sampling Depth and Alteration of Soil Processes.” *Soil Science Society of America Journal* 77, no. 5 (2013): 1818–24. https://doi.org/10.2136/sssaj2013.01.0022.

Tasissa, G, H E Burkhart, and R L Amateis. “Volume and Taper Equations for Thinned and Unthinned Loblolly Pine Trees in Cutover, Site-Prepared Plantations.” *Southern Journal of Applied Forestry* 21, no. 3 (1997): 146–52. https://doi.org/10.1093/sjaf/21.3.146.

Thomson, B D, T S Grove, N Malajczuk, and G E Hardy. “The Effect of Soil PH on the Ability of Ectomycorrhizal Fungi to Increase the Growth of Eucalyptus Globulus Labill.” *Plant and Soil* 178, no. 2 (1996): 209–14. https://doi.org/10.1007/bf00011585.

Thomas, G W. “Soil PH and Soil Acidity.” Essay. In *Methods of Soil Analysis*, 475–90. 5. Soil Science Society of America, 1996.

Tiedemann, A R, R R Mason, and B E Wickman. “Forest Floor and Soil Nutrients Five Years After Urea Fertilization in a Grand Fir Forest.” *Northwest Science* 72, no. 2 (May 1998): 88–95.

Tschaplinski, T J, R J Norby, D E Todd, and D W Johnson. “Biomass and SOIL NITROGEN Relationships of a One-Year-Old SYCAMORE PLANTATION.” *Soil Science Society of America Journal* 55, no. 3 (1991): 841–47. https://doi.org/10.2136/sssaj1991.03615995005500030036x.

USDA NRCS, Soil quality indicators: pH § (1998).

Vogel, J G, and E J Jokela. “Micronutrient Limitations in Two Managed Southern Pine Stands Planted on Florida Spodosols.” *Forest, Range & Wildland Soils* 75, no. 3 (May 1, 2011): 1117–24. https://doi.org/ https://doi-org.prox.lib.ncsu.edu/10.2136/sssaj2010.0312.

Weaver, A R, D E Kissel, F Chen, L T West, W Adkins, D Rickman, and J C Luvall. “Mapping Soil PH Buffering Capacity of Selected Fields in the Coastal Plain.” *Soil Science Society of America Journal* 68, no. 2 (2004): 662–68. https://doi.org/10.2136/sssaj2004.6620.

Welegedara, N P, R F Grant, S A Quideau, and S Das Gupta. “Modelling Nitrogen Mineralization and Plant Nitrogen Uptake as Affected by RECLAMATION COVER Depth in Reclaimed Upland Forestlands of Northern Alberta.” *Biogeochemistry* 149, no. 3 (May 25, 2020): 293–315. https://doi.org/10.1007/s10533-020-00676-5.

Zhao, and Jing. “Effect of Annual Variation in Soil PH on Available Soil Nutrients in Pear Orchards.” *Acta Exologica Sinica* 31, no. 4 (2011): 212–16.

Chapter 2: The Macronutrient Calcium and its effects on Loblolly Pine (*Pinus taeda*) Growth and Stem Sinuosity in Organic Soil

Introduction

Loblolly pine (*Pinus taeda*) is one of the most economically important trees in the world today and demand for loblolly pine products is projected to grow by 30-40%. Loblolly pine is used to produce pulpwood, plywood, and a wide range of other different products. Loblolly pine makes up 12% of paper and 19% solid wood products globally (Oswalt 2012). To meet the demand for wood products, companies and landowners grow loblolly pines commercially in heavily managed plantations to achieve optimal growth. Organic soils have been of interest for loblolly pine management since the 1970's due to relatively high productivity potential with artificial drainage (MacCarthy and Davey, 1976). In the United States, it is estimated that four million ha exist in the southeast within the natural range of loblolly pine.

While research has shown that nitrogen (N) and phosphorous (P) are the two largest growth limiting factors in loblolly pine (Carlson et al. 2013, Fox et al. 2007, Albaugh et al. 2018, Albaugh et al. 1998), organic soils are high in N content and typically low in pH due to the high organic matter content of at least 20-30% in the first 40 cm from the top (NCSS 2008). Loblolly pine grows naturally in soil pH range is 4.5 to 6. Soil pH is closely tied to cation exchange capacity and base saturation of the soil. In agriculture, Ca fertilizer such as lime is used to increase the soil pH and add Ca as a nutrient (Thompson et al. 2011). MacCarthy and Davey (1976) showed an increase of height with the addition of limestone and P. Kyle et al (2005) showed an increase of volume growth with the addition of P+N and limestone.

Unfortunately, while overall productivity of loblolly pines on organic soil can be quite high, a growth deformity known as stem sinuosity can be common (Espinoza et al. 2012). Stem

sinuosity is defined as “any stem crookedness that occurs in the segment between two whorls” (Campbell, 1965) resulting in slight to severe curvature affecting the stem quality (Espinoza et al., 2012). Stem sinuosity effects many trees such as loblolly pine (*Pinus taeda*), and many other tree species such as radiata pine (*Pinus radiata*) and douglas fir (*Pseudotsuga menziesii*). It is not precisely known what causes stem sinuosity in trees, but could connected to a nutritional imbalance, genetic predisposition, previous land use, or a combination of the three (Spicer et al. 2000 and Hopmans et al., 1995). In radiata pine, the syndrome has been associated with previous land use and high-N levels (Hopmans et al., 1995). In 1991, Birk found stem deformities correlated with foliar manganese (Mn), aluminum (Al), and calcium (Ca) in radiata pine. Espinoza et al (2012) found N increased stem sinuosity in loblolly pine, whereas Ca decreased sinuous stems. Jones and Fox (2012) found a growth related response in juvenile loblolly pines, where less sinuous trees had smaller diameter breast height (DBH) than trees that were more sinuous.

Ca is an essential nutrient that is responsible for the structure of cell walls and membranes (White and Broadley 2003), thus Ca is vital in wood formation. Ca has a great ability to form inter- and intra- molecular co-ordination complexes to link and modify structures within plants (Clarkson 1984). A tree experiencing Ca deficiencies may show disintegrated cell walls leading to the collapse of petioles and upper parts of the stem (Marschner 1995). Previous studies have pointed to nutrient deficiencies within loblolly pine, such as copper (Cu) and calcium (Ca) (South et al. 2004, Kyle et al 2005, MacCarthy and Davey 1976).

In this study, we examine the impact of a calcium carbonate fertilizer on the relative growth and form of loblolly pine on organic soils. Our objectives are to evaluate i) growth response and stem form due to source and rate of Ca fertilizer, ii) genetic interactions with

source and rate of Ca fertilizer application, and iii) how application rate and source affect soil nutrients and foliar nutrition (field) and soil pH (field and lab) over time. We hypothesize that 1) Calciprill and dolomitic limestone will have comparable influences on tree growth and stem form, 2) application rates increase, growth response will increase and stem form quality will improve, 3) some genetics will have greater improvements in stem form than others with the application of calcium, and 4) increased rates will increase soil and foliar Ca content and raise soil pH, making other soil nutrients more available.

Methods and Materials

Study Sites

For this project, two study sites were established in 2018. One site was located in Hofmann Forest in Jones County, North Carolina (Hof2018) and the other in Brunswick County, North Carolina (Brun2018; Figure 2.1). Soils are the same series: Croatan (loamy, siliceous, dysic, thermic Terric Haplosaprists). Details of site code, establishment age, establishment year, and location in Table 2.1. Three additional sites were established between 2019 and 2021 but have not yet matured for measurements (for details see Appendix A).

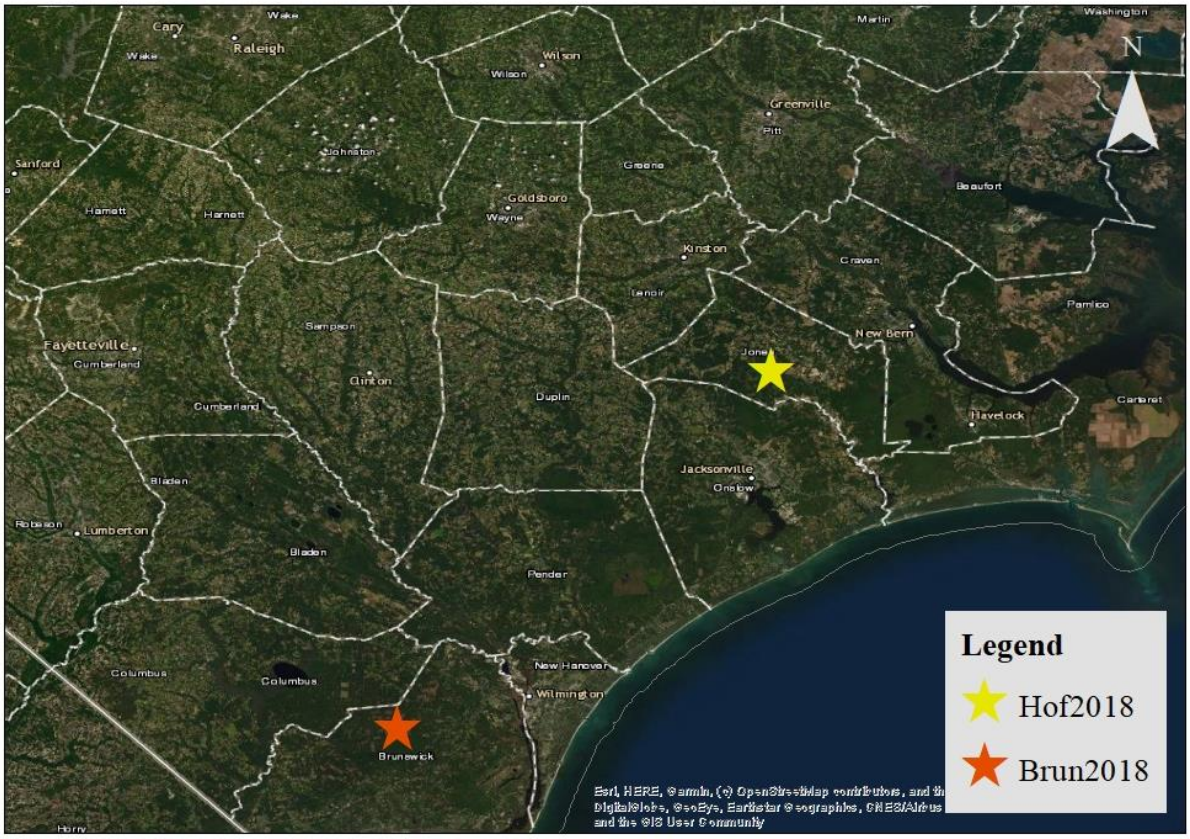


Figure 2.1. Map of southeastern North Carolina with study site locations

Table 2.1. Site code, site name, establishment date, location, and pretreatment soil pH, Ca, P and 0.5 years since treatment (YST) N.

Site Code	Site Name	Est Date	Age at Est (yrs)	Location	YST 0 Soil pH	YST 0 Ca in soil (ppm)	YST 0.5 N in soil (%)	YST 0 P in soil (ppm)
994001	Hof2018	Feb 2018	1	Jones County, NC	3.77	117	0.94	23.98
994002	Brun2018	Fed 2018	1	Brunswick County, NC	3.37	191	1.09	9.77

Experimental Design

Study sites Hof2018 and Brun2018 are arranged in a split plot design with three replicates at each site blocked by initial tree height and root collar diameter measurements. Treatment plots are comprised of 108 trees (9 rows of 12 trees). Measurement plots are centered in the treatment plot and comprise 30 trees (5 rows of 6 trees). Measurement plot size is 0.03 ha. Each site study area is 1.3 ha. All treatments had complete weed control, a combination of chemical site preparation and herbaceous weed control with directed sprays of sulfometuron methyl and glyphosate, and all trees received fipronil for pine tip moth control. All treatments received 10 kg ha^{-1} elemental phosphorous (P) as 2 oz. triple super phosphate (20% elemental P) per tree.

Tree height and bed height measurements were taken prior to treatment assignment. Height was measured with a meter stick from base of tree to top of tree. Bed height was taken with two meter sticks, one with a level attached in the center. The meter stick with level attached was placed a base of tree horizontally and the second meter stick was positioned vertically at base of tree bed so that the two yardsticks intersected each other (Appendix A). Measurements were taken when the first meter stick was level. Measurements were taken for every tree in the measurement plot and used to help assign blocking for fertilization treatments.

At both Hof2018 and Brun2018 sites (Figures 2.2 & 2.3, respectively), there were four treatments consisting of a control (no Ca), 400 kg ha^{-1} Ca as Calciprill, 800 kg ha^{-1} Ca as Calciprill, and 800 kg ha^{-1} Ca as Dolomitic Lime (see Table 2 for all site treatments) that served as the whole plot treatment. Split plots within each treatment at sites Hof2018 and Brun2018 consisted of five genetics per plot with one row each: AGM-22, AGM-37, AG-783, AG-373, and AG-786. Additional sites established to continue and expand the study were installed in 2020

and 2021. Details on experimental design can be found in Appendix B.

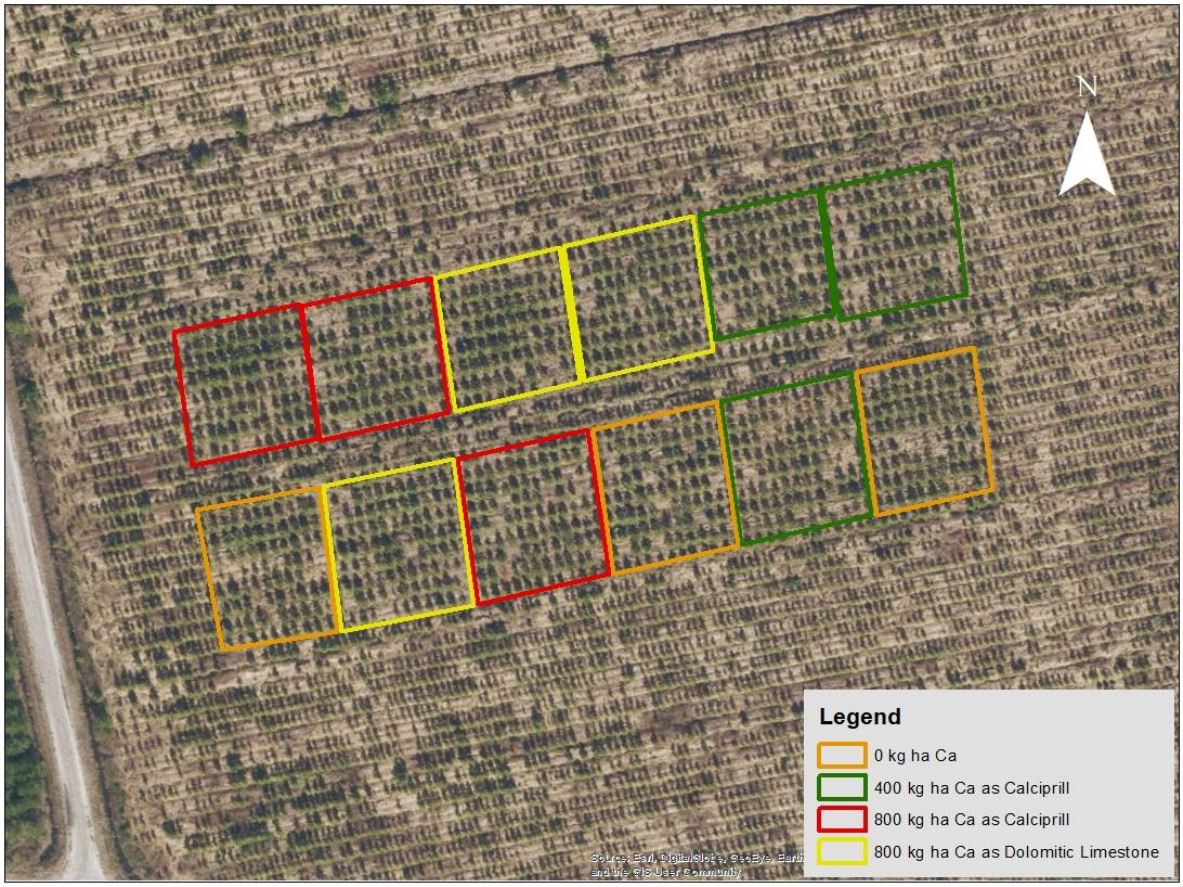


Figure 2.2. Site Hof2018 map with treatment plots and corresponding treatment descriptions.

Figure 2.3. Site Brun2018 map with treatment plots and corresponding treatment descriptions.

Tree Measurements

Measurements were conducted each year during the winter months between December – February while trees are dormant. Height, diameter breast height (DBH), sweep, height to live crown (HTLC) and sinuosity were taken for every tree in the measurement plots. Height was measured with a height pole placed at base of tree and raised until pole is on a level with top of tree. DBH taken a breast height (~1.2 meters) off ground with a DBH tape or calipers. Stem straightness, or sweep, was measured as the maximum deflection (nearest 1 cm) from a 3 m pole set against the vertical axis of the tree (Appendix C). A straight edge is held next to the tree and moved up along the first log, until the 3 m pole reaches a height of approximately 5 m. The maximum deflection measured in 1 cm increments. HTLC was measured with a meter stick at base of tree; measurement taken at the first living branch on the stem. Sinuosity was taken as a categorical stem straightness rating on a scale of 1-3 for the top third of tree stem. Rating of "1" meant stem deemed straight. Rating of "2" meant stem was not straight. Rating of "3" meant stem was "S" shaped (Figure 2.4).

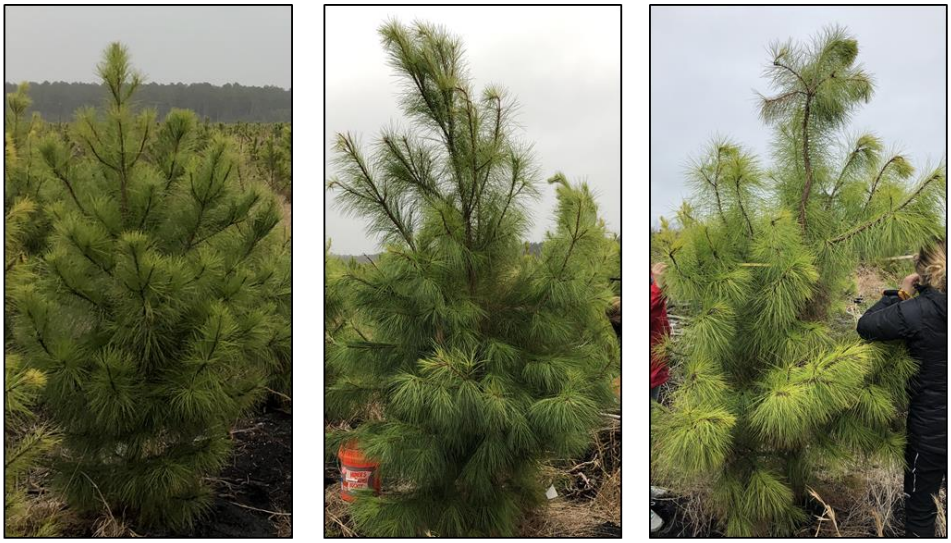


Figure 2.4. Example of the tree-sinuosity ratings.

Soil Sampling

Surface soil was sampled and composited from five locations in the interbed and five locations in the bed in the measurement plot with a soil auger (6 cm diameter) at depth increments 0-30 cm. Samples were air-dried and ground with soil grinder, then passed through a 2 mm sieve.

Soil samples were taken before fertilization with initial tree measurements. Soil sampling also occurs six months and year one. Initial soil analyses and subsequent soil sampling include extractable P, K, Ca, Mg (Mehlich III extraction), and pH (Appendix E). All soil analyses completed by Waters Ag Lab.

Foliage Sampling

Foliage collection occurred at the time of tree measurements at year 0 and one-year post fertilization. Samples were collected from five dominant trees in each measurement plot. Pine needles were collected from the uppermost laterals in the upper third of the live crown. Each sample comprised of 100 fascicles collected from the five sample trees. Foliage was cleaned of contaminates and then oven dried. Samples analyzed by Waters Agricultural Lab for the ICP test, which tests nitrogen, phosphorus, potassium, magnesium, calcium, sulfur, boron, zinc, manganese, iron and copper (Appendix D).

Soil pH Incubation Experiment

Soil was collected with a clean spade in bulk from site Hof2020 in the spring of 2020 from the first 0-15 cm. In the laboratory, soil was placed into trays and air-dried until weight was consistent. Air dried soil was ground and passed through a 2 mm sieve. Soil that did not pass through a 2 mm sieve was discarded.

The calcium sources for the incubation were highly reactive Ca as Calciprill ($CaCO_3$, 37% elemental Ca), and dolomitic lime ($CaMg(CO_3)_2$, 23% elemental Ca). Calciprill granules were ground with a mortar and pestle to pass through a 2 mm sieve. Fragments that did not pass through the sieve were discarded. Dolomitic lime was procured as a very fine powder and all passed a 2 mm sieve.

Soil (150 g dry weight) was placed in 473 ml glass canning jars. Jars were topped with the lids drilled with a 1 cm hole for gas exchanged. Jars were randomly assigned treatments and number of days of incubation. Each treatment was replicated three times for each incubation period. The incubation periods were 0, 1, 2, 4, 7, 14, 21, and 35 days. Appropriate amounts of fertilizer were measured out for each treatment (Table 3) and mixed with soil based on jar surface area.

Table 2.2 Soil pH lab incubation experiment. Treatment descriptions and total fertilizer used per jar.

Treatment Description	Total Fertilizer per Jar (g)
0 kg ha^{-1} Ca	0
400 kg ha^{-1} Ca as Calciprill	0.63
800 kg ha^{-1} Ca as Calciprill	1.25
800 kg ha^{-1} Ca as Dolomitic Lime	2
1600 kg ha^{-1} Ca as Calciprill	2.5
3200 kg ha^{-1} Ca as Calciprill	5

Deionized Water was then added to each jar to bring the moisture content to 80-90% of field capacity. Field capacity was determined gravimetrically for three samples by placing soil in

a funnel lined with filter paper over a graduated cylinder. Equal amounts of water were poured over each sample. Once water stopped dripping out of each sample, the amount of water collected in each cylinder was recorded. To track water content, jars were weighed every 7 days during incubation, and deionized water was added to keep soil moisture constant.

After incubation time, soil was removed from jar and oven dried at 40^{o}C, then ground with mortar and pestle to break up larger clumps of soil, then passed through a 2 mm sieve. Soil was mixed in a 2:1 ratio with 0.01-mol L $CaCl_2$ solution to minimize the effects of salt concentrations that affect soil pH. Soil pH was determined by an Accumet Model-25 pH meter.

Statistics

Response variables for the field studies were cumulative height and DBH, sinuosity, HTLC. All response variables shown are from 3 years after treatment. Soil pH, nutrient content in soil, and nutrient content in foliage were analyzed at 0.5 and 1-year post treatment application to assess changes post treatment. The fixed whole-plot effect in this study was treatment (application rate and source of Ca fertilizer applied) with genetics nested within as the split plot. Block was treated as a random effect. Site x treatment interactions were analyzed and where significant, sites were analyzed separately.

The response variable for the incubation study was soil pH with Ca source and rate treatments as fixed effects. Responses to treatments in the incubation experiment were analyzed per time period. Alpha level was set at 0.05 for all significance tests. Differences among treatments were tested using the Tukey's separation of means on JMP version 15 (JMP, Version 15. SAS Institute Inc., Cary, NC, 1989-2021).

Results

Mortality

Both sites were hit in the fall of 2018 by hurricane Michael. In 2018, mortality was comparable between Hof2018 (4%) and Brun2018 (X%, Figure 2.6). After the hurricane, Brun2018 has significantly more mortality with X% compared to Hof2018 ($p<0.01$). Hof2018, initially after the hurricane did not see a significant amount of mortality, but as of 2021 the mortality rate is significantly more than 2018 and 2019 (Figure 2.5) ($p<0.01$). In 2021, Hof2018 has a 4% mortality level. Brun2018 experienced an increase in the mortality rate up to 26% in 2021 ($p<0.01$).

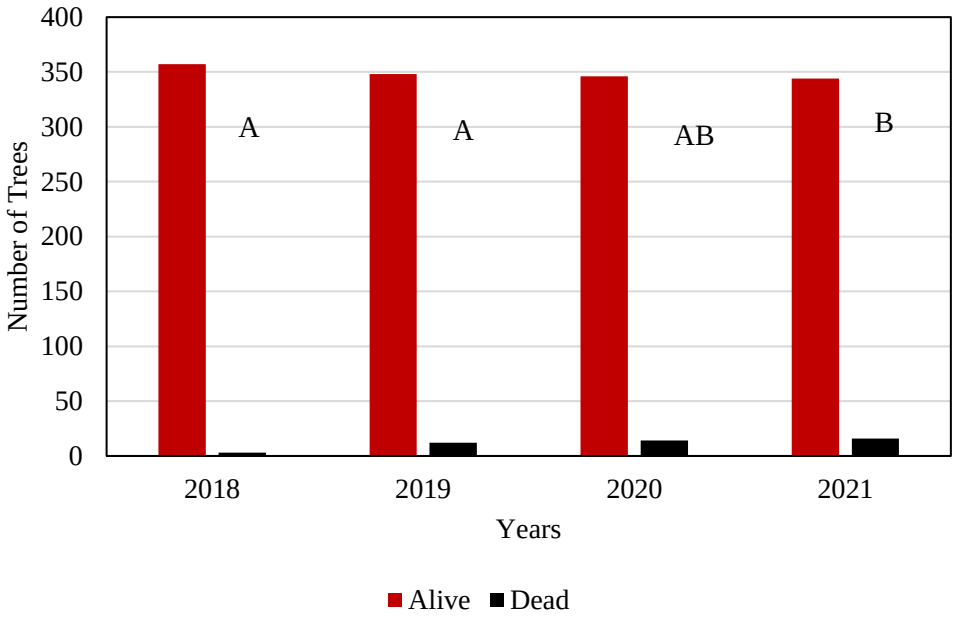


Figure 2.5. Hof2018 mortality by year. Year 2021 mortality is more significant than years 2018 and 2019 ($p=0.0099$). Treatments that have similar letter, or groups with no letters, are not significantly different at $p = 0.05$ significance level.

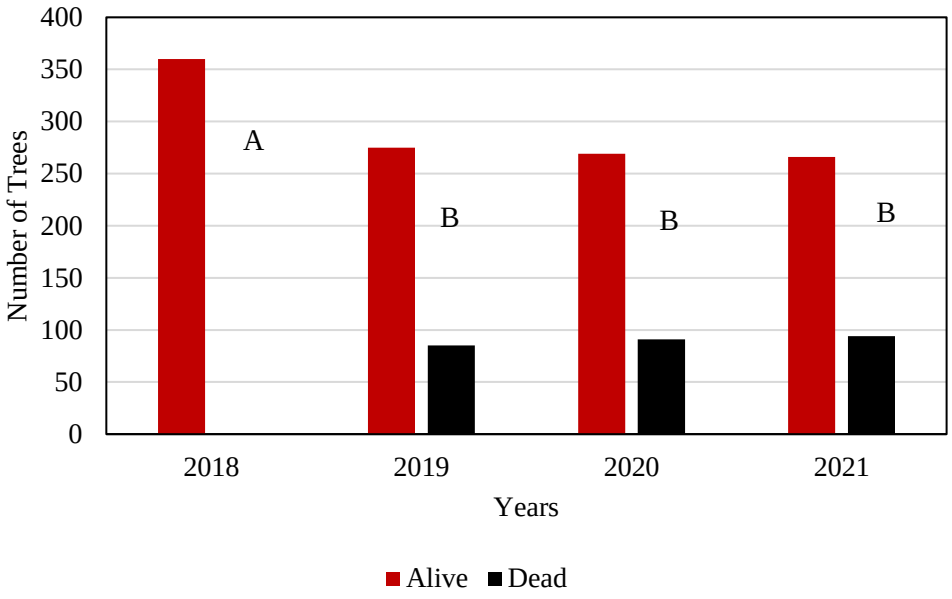


Figure 2.6. Brun2018 mortality by year. Years 2019, 2020 and 2021 mortality is more significant than year 2018 ($p<0.0001$). Treatments that have similar letter, or groups with no letters, are not significantly different at $p = 0.05$ significance level.

Tree Growth

At the Hof2018 and Brun2018 sites in years one and two since treatment, we found no difference due to treatment, genetics, or interaction for height, DBH, or volume. In year 3, heights showed different results due to treatment between study sites. Hof 2018 showed a significant effect due to treatments (Figure 2.7), while Brun2018 showed no differences (Figure 2.8). At Hof2018, the 400 kg ha^{-1} Ca as Calciprill treatment heights were shorter than the other treatments, which were all similar. Hof2018 showed a significant effect in interaction with genetics and height (Figure 2.9). Genetic AG-783 with treatment 400 kg ha^{-1} Ca as Calciprill being statistically shorter than trees of AGM-22 in treatment 800 kg ha^{-1} Ca as Calciprill and the rest of the genetics fell in-between.

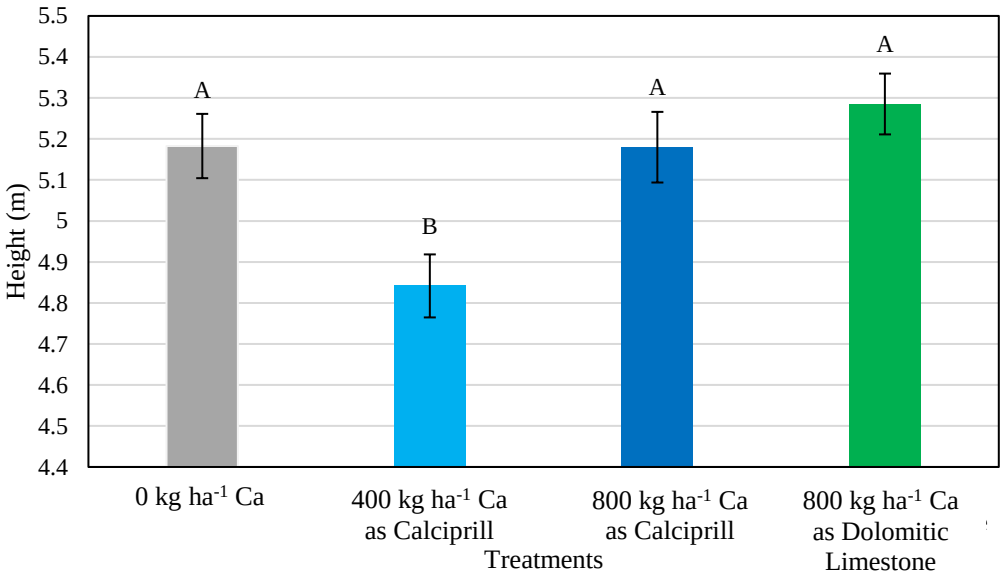


Figure 2.7. Year 3 height data from Hof2018 by treatment (p <<=<<= 0.0005). Error bars represent standard error of the mean. Treatments that have similar letter, or groups with no letters, are not significantly different at p = 0.05 significance level.

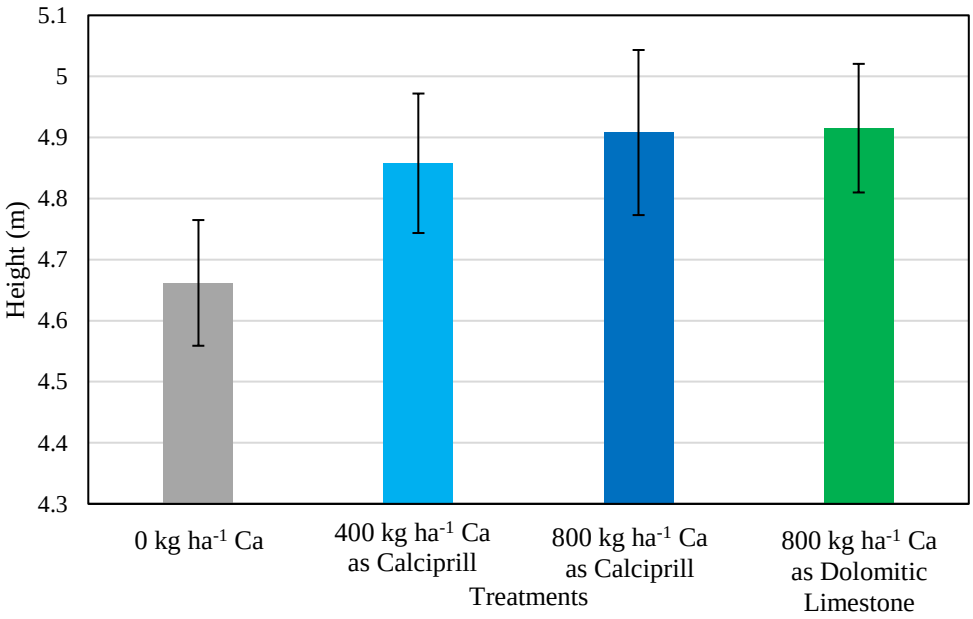


Figure 2.8. Year 3 height data from Brun2018 by treatments (p = 0.357). Error bars represent standard error of the mean.

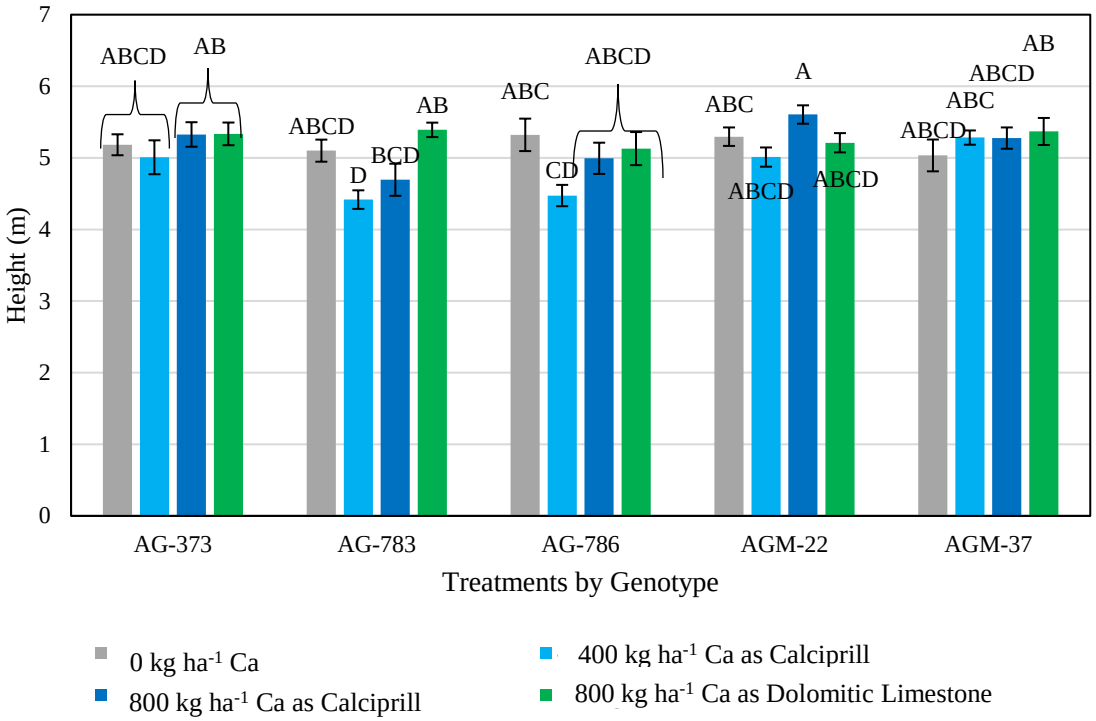


Figure 2.9. Year 3 height data from sites Hof2018 by genetics and treatment. Significant at p = 0.0003. Error bars represent standard error of the model. Treatments that have similar letter, or groups with no letters, are not significantly different at p = 0.05 significance level.

Three years after treatment, DBH differed among treatments at site Hof2018 (Figure 2.10). The 800 kg ha^{-1} Ca as Calciprill was about 0.75 cm greater than the 0 kg ha^{-1} Ca and 400 kg ha^{-1} Ca as Calciprill treatments, 800 kg ha^{-1} Ca as Dolomitic Limestone was comparable to all three treatments. Brun2018 showed no differences between treatments for DBH (Figure 2.11). Hof2018 showed a significant effect in interaction with genetics and DBH (Figure 2.12). Genetic AG-783 with treatment 400 kg ha^{-1} Ca as Calciprill DBH being statistically smaller than DBH of trees AGM-22 in treatment 800 kg ha^{-1} Ca as Calciprill and the rest of the genetics fell in-between.

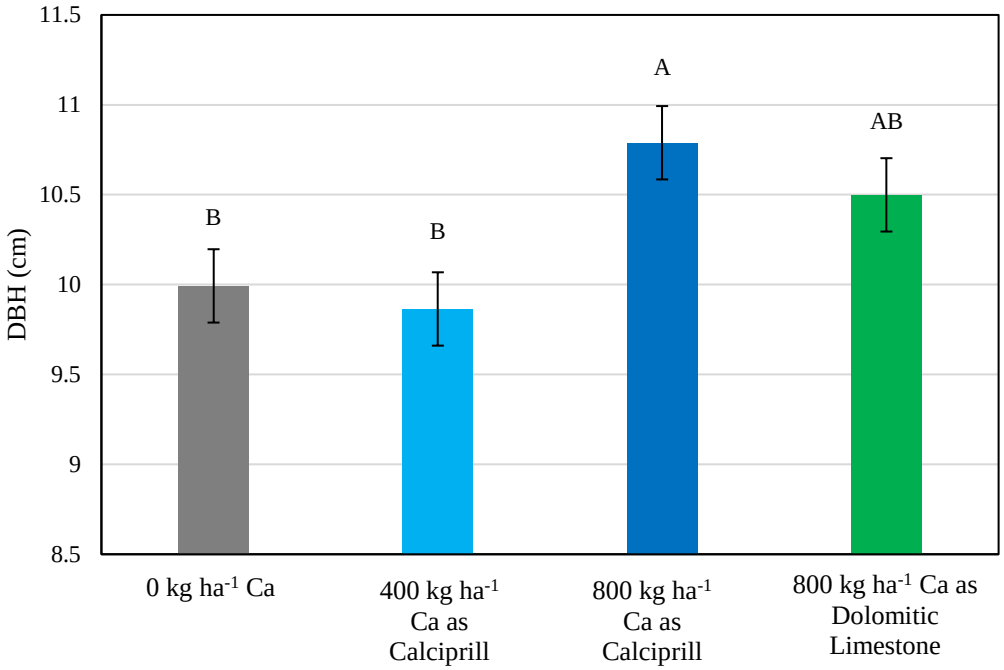


Figure 2.10. Year 3 DBH data for sites Hof2018. Significant at p = 0.0033. Error bars represent standard error of the model. Treatments that have similar letter, or groups with no letters, are not significantly different at p = 0.05 significance level.

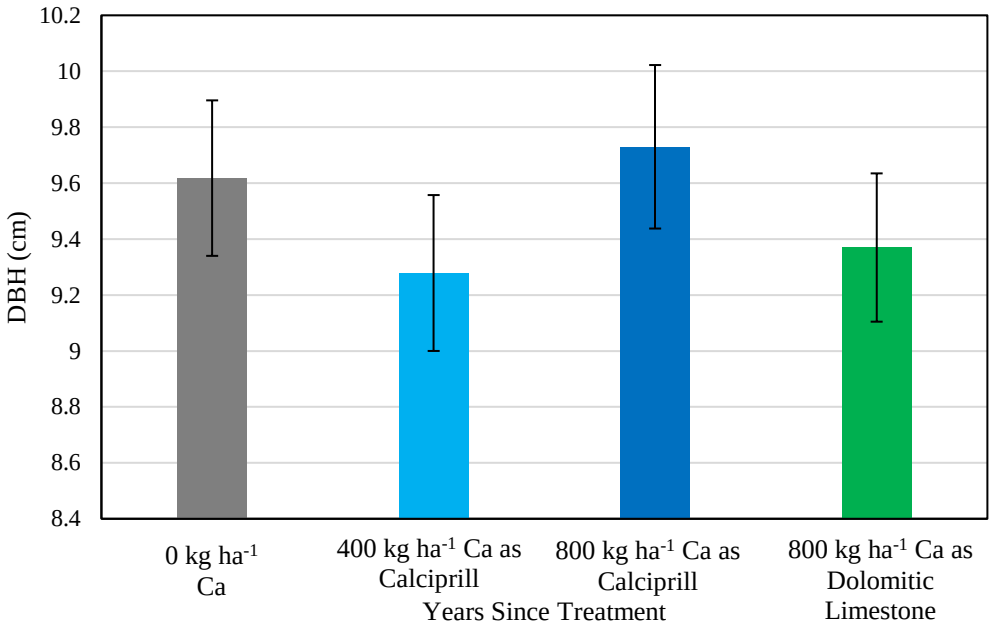


Figure 2.11. Year 3 DBH data for sites Brun2018. Not significant at p = 0.6453. Error bars represent standard error of the model.

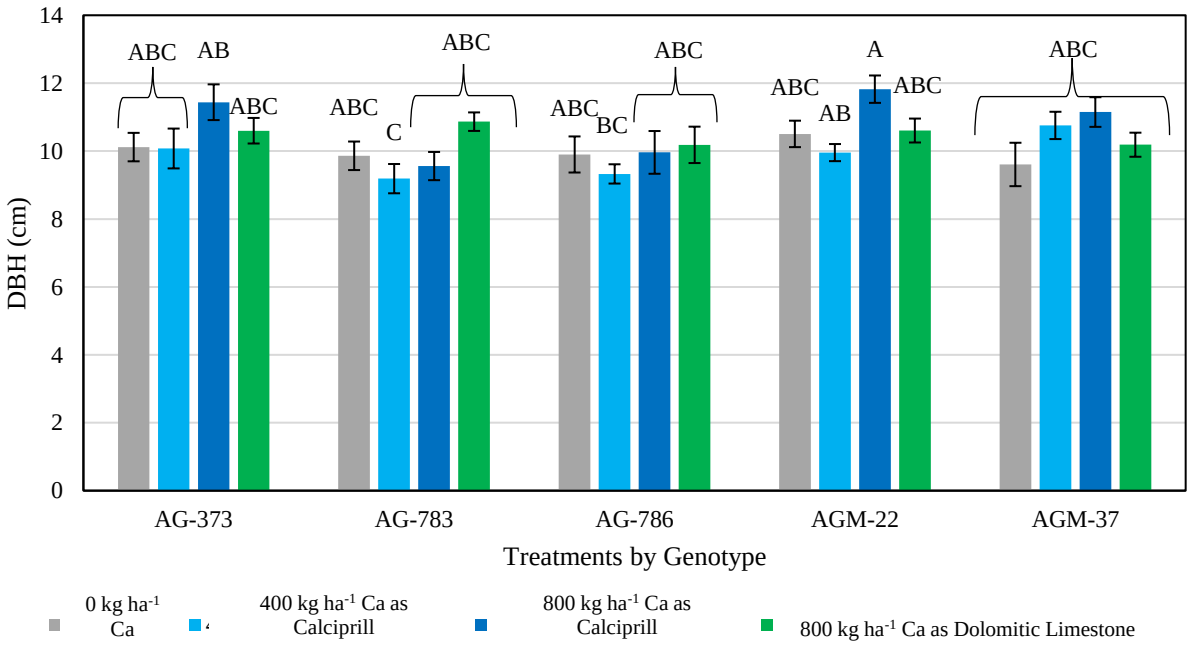


Figure 2.12. Year three DBH data from site Hof2018 by genotypes and treatments. Significant at p = 0.0074. Error bars represent standard error of the model. Treatments that have similar letter, or groups with no letters, are not significantly different at p = 0.05 significance level.

Three years after treatment, volume differed among treatments at site Hof2018 (Figure 2.13). The 800 kg ha^{-1} Ca as Calciprill tree volume was greater than the 0 kg ha^{-1} Ca and 400 kg ha^{-1} Ca as Calciprill treatments but comparable to 800 kg ha^{-1} Ca as Dolomitic Limestone. Treatment 800 kg ha^{-1} Ca as Dolomitic Limestone was comparable to 0 kg ha^{-1} Ca, but not to 400 kg ha^{-1} Ca as Calciprill. Brun2018 showed no differences between treatments for volume (Figure 2.14). Hof2018 showed a significant effect in interaction with genetics and volume (Figure 2.15). Genetics AG-783 and AG-786 with treatment 400 kg ha^{-1} Ca as Calciprill volumes being statistically smaller than volume of trees AGM-22 in treatment 800 kg ha^{-1} Ca as Calciprill and the rest of the genetics fell in-between.

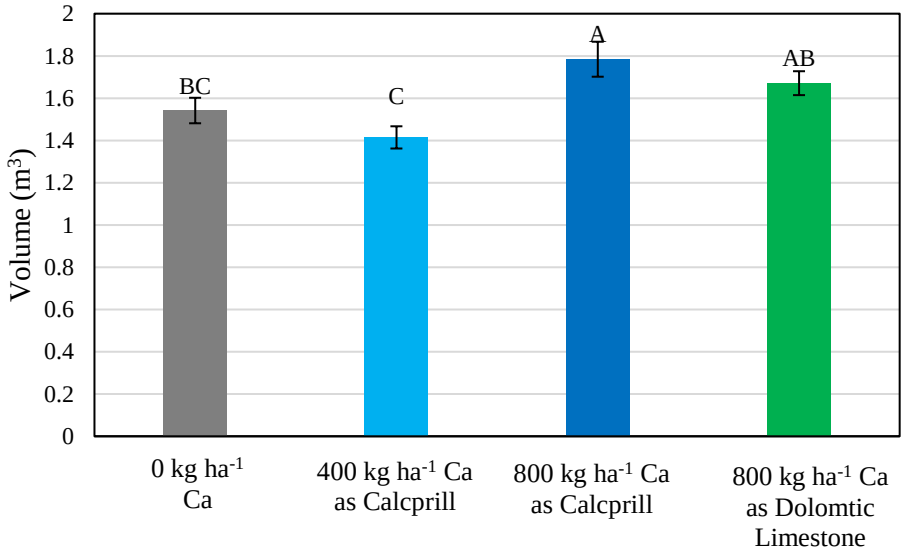


Figure 2.13. Year 3 volume data for site Hof2018. Significant at p = 0.0004. Error bars represent standard error of the model. Treatments that have similar letter, or groups with no letters, are not significantly different at p = 0.05 significance level.

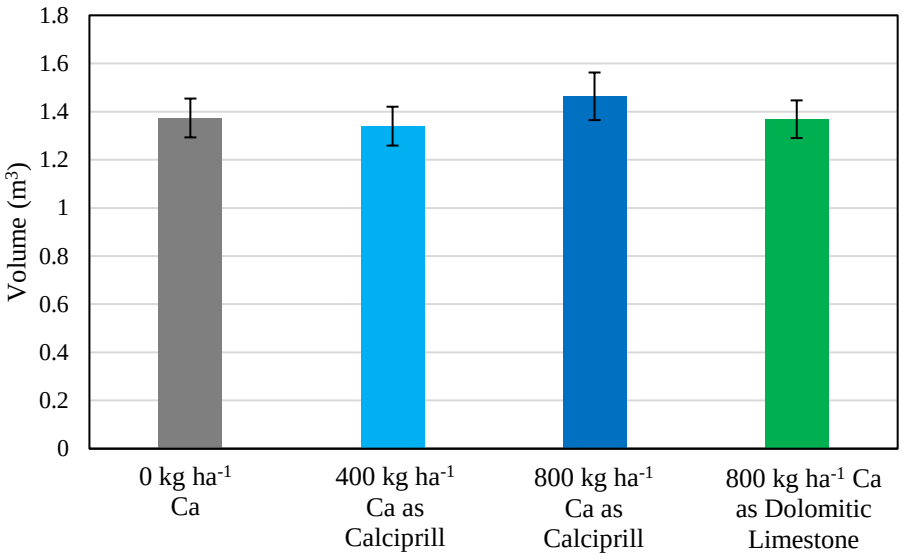


Figure 2.14. Year 3 volume data for site Brun2018. Not significant at p = 0.7654. Error bars represent standard error of the model.

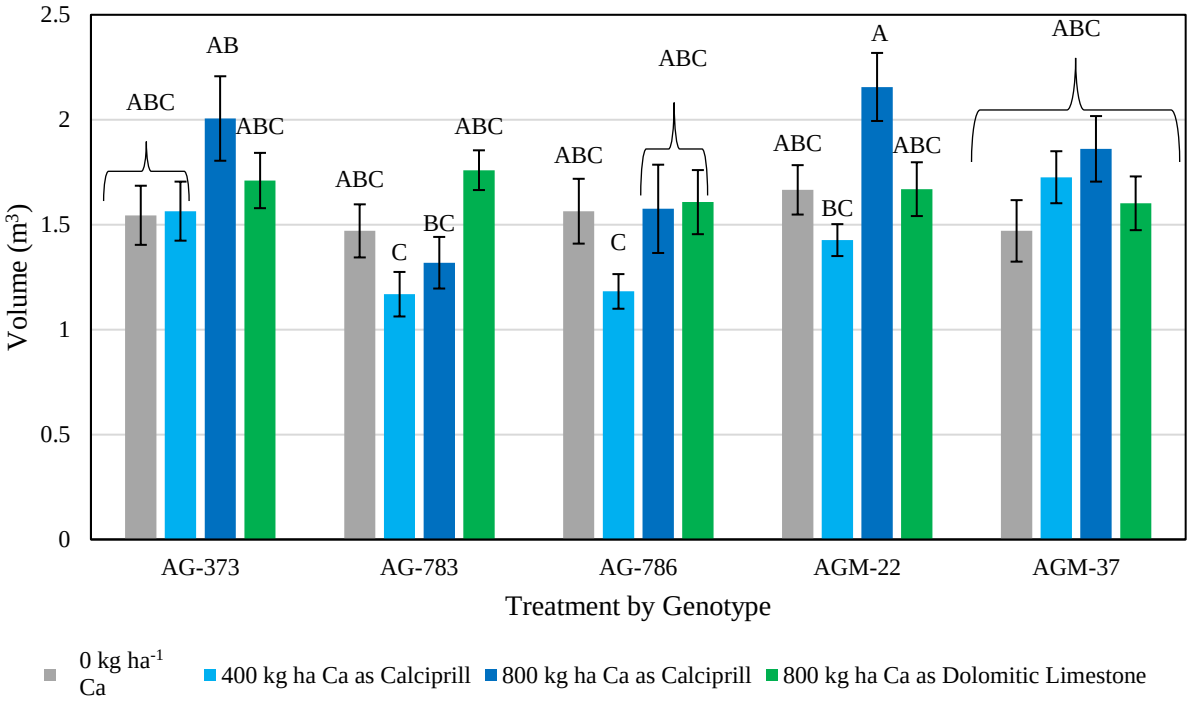


Figure 2.15. Year three volume data from site Hof2018 by genotypes and treatments. Significant at p = 0.0004. Error bars represent standard error of the model. Treatments that have similar letter, or groups with no letters, are not significantly different at p = 0.05 significance level.

Site Hof2018 had no significant differences between treatments for sinuosity ($p = 0.343$) (Figure 2.14). At Brun2018, treatment 400 kg ha^{-1} Ca as Calciprill trees were less sinuous than treatments 0 kg ha^{-1} Ca trees and 800 kg ha^{-1} Ca as Dolomitic Limestone, with treatment 800 kg ha^{-1} Ca as Calciprill falling in-between (Figure 2.15). There were no significant differences between the genetics and treatment concerning the sinousity rating at either site or together. At both sites sinuosity ratings between different genetics were the same, with genotype AG-783 have more sinuous trees than genotypes AG-373, AGM-22 and AGM-37 ($p < 0.0001$) (Figure 2.16). Hof2018 had significant differences between treatments for HTLC measurement, where treatment 800 kg ha^{-1} Ca as Calciprill had the least amount of HTLC (HTLC = 0.36) and 0 kg ha^{-1} Ca had the most amount of HTLC (HTLC = 0.44), with 400 kg ha^{-1} Ca as Calciprill and 800 kg ha^{-1} Ca as Dolomitic Limestone treatments were comparable to both ($p = 0.0365$) (Figure 2.17). There were no detectable differences in HTLC at Brun2018 ($p = 0.02525$) (Figure 2.18).

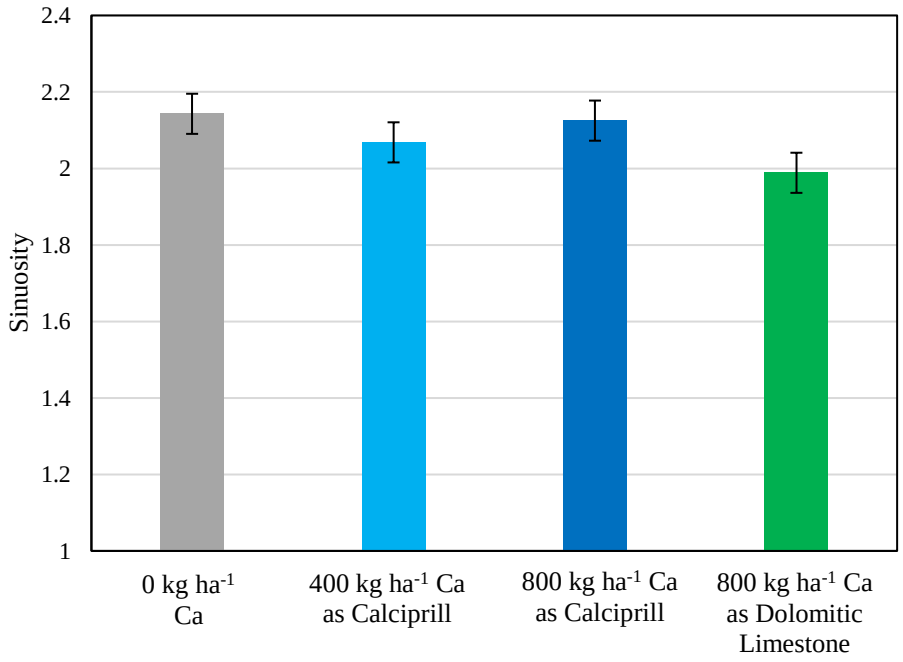


Figure 2.16. Year 3 sinuosity data from Hof2018 by treatment descriptions. No significant differences between treatments at $p = 0.343$. Error bars represent standard error of the model.

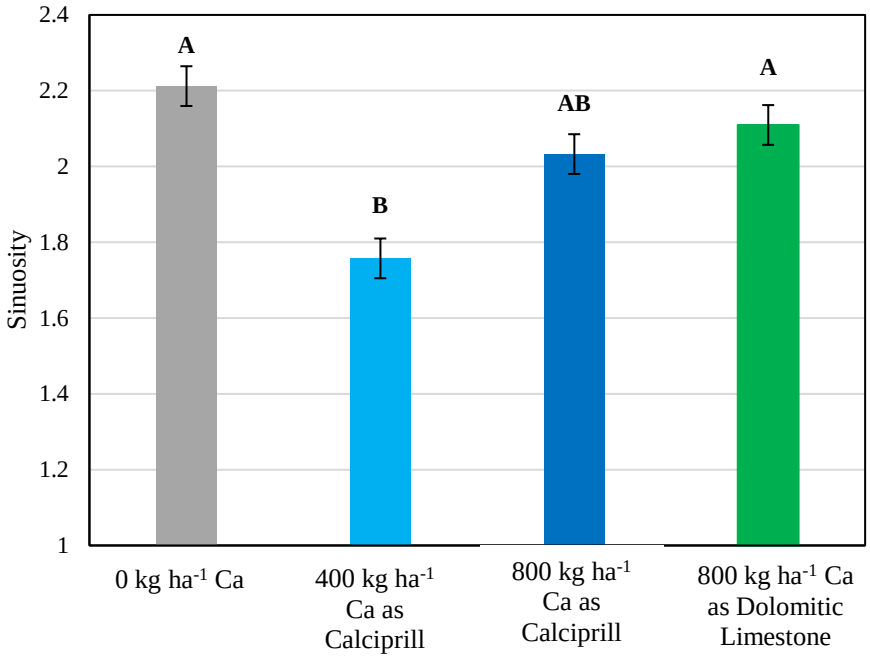


Figure 2.17. Year 3 sinuosity data from Brun2018 by treatment descriptions. Different letters are significant at p = 0.0042. Error bars represent standard error of the model.

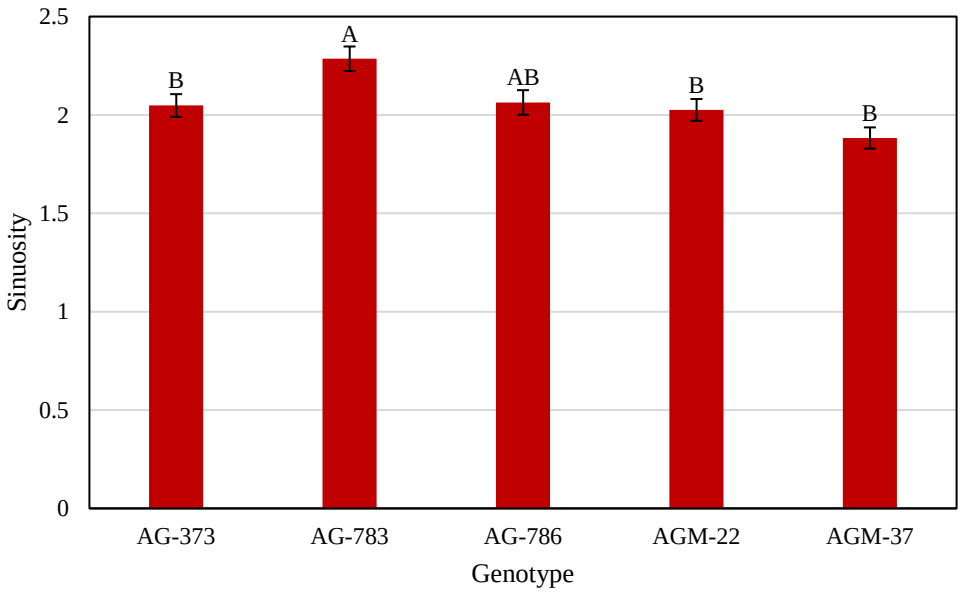


Figure 2.18. Year 3 sinousity data from sites Hof2018 and Brun2018 by genotype. Different letters are significant at p < 0.001. Error bars represent standard error of the model.

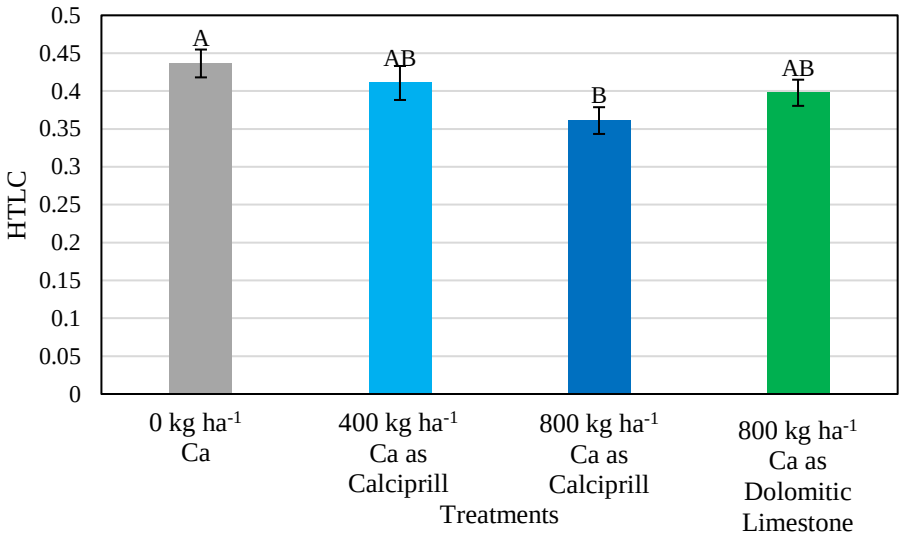


Figure 2.19. Year 3 HTLC data from Hof2018 by treatment descriptions. Different letters are significant at p = 0.0365. Error bars represent standard error of the model.

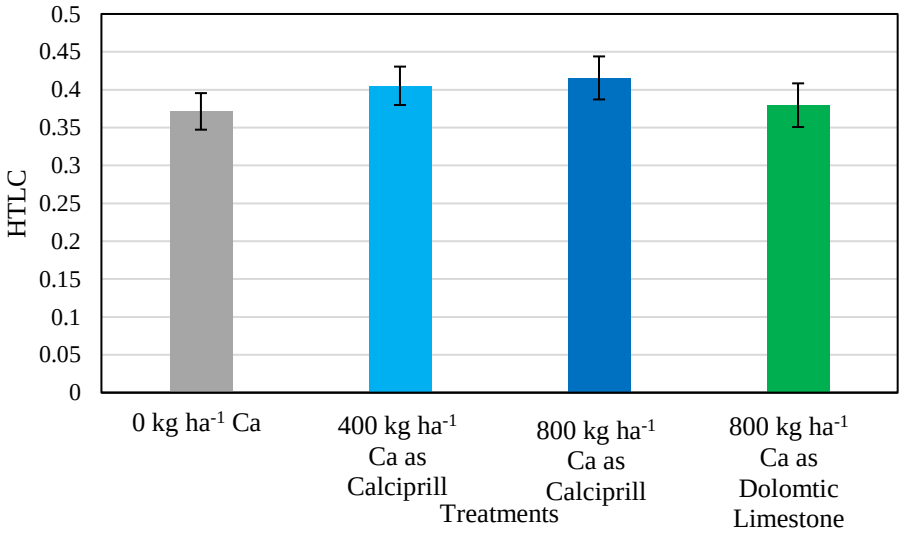


Figure 2.20. Year 3 HTLC data from Brun2018 by treatment descriptions. No significant differences at p = 0.2525. Error bars represent standard error of the model.

Field Nutrient Data

Soil pH before fertilization was at the two sites were significant different from each other. Hof2018 before treatment soil pH was 3.75. Hof2018 year one treatment soil pH were not significant different from one another (p=0.3433), but year one soil pH was significantly higher than before treatment as a whole (p=0.0031) (Figure 2.19). Brun2018 before treatment soil pH was 3.4. Hof2018 year one treatment soil pH were not significant different from one another (p=0.0504), but year one soil pH was significantly higher than before treatment as a whole (p<0.0001) (Figure 2.20).

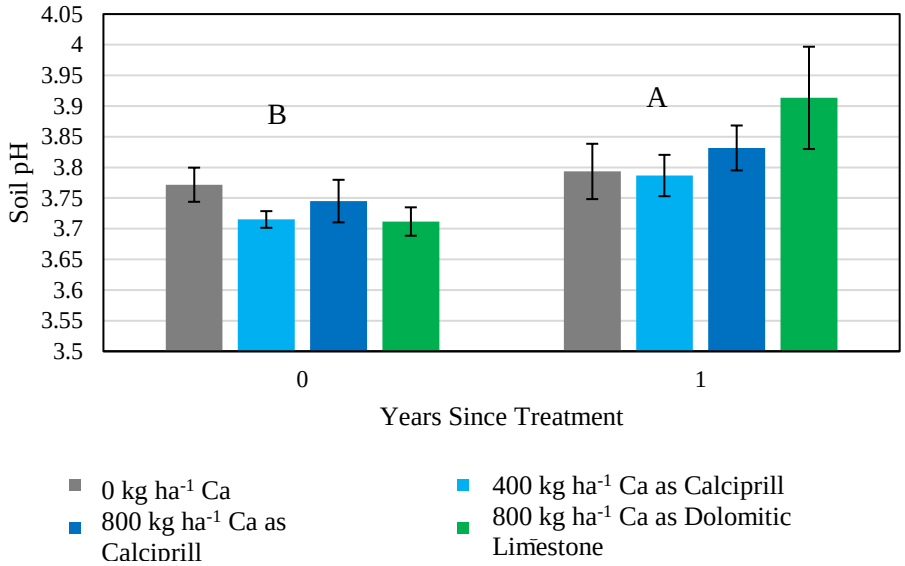


Figure 2.21. Hof2018Year 0 and 1 since treatment soil pH data by treatment description. Year 1 soil pH significantly higher than year 0 at p = 0.0027. Treatments within each period were not statistically different from one another at p =0.3433. Error bars represent standard error of the model.

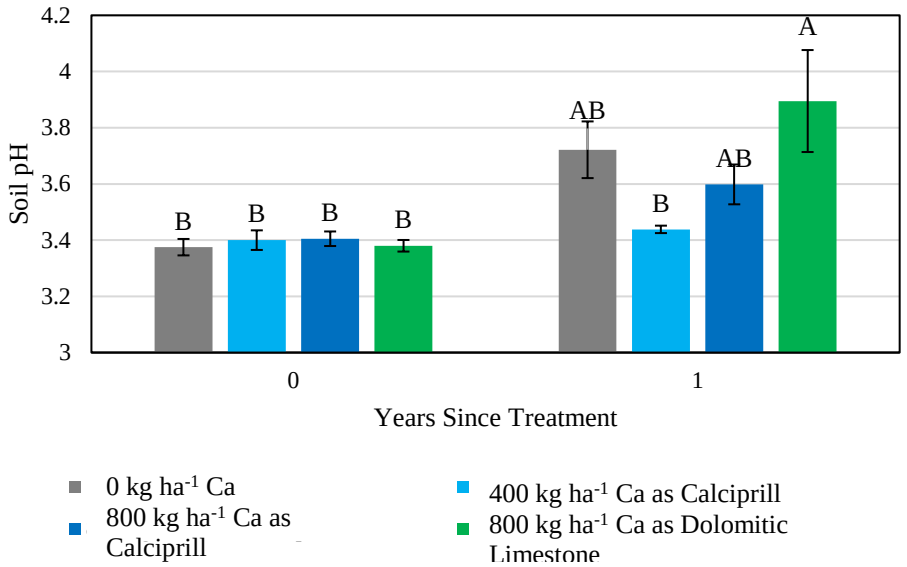


Figure 2.22. Brun2018 Year 0 and 1 since treatment soil pH data by treatment description. All treatments and YST analyzed together. Treatments with different letters are statistically different at p = 0.0187 and those with similar letters are not different at p =0.05. Error bars represent standard error of the model.

Calcium content in the soil was significantly increased 6 months and one-year post fertilization from where calcium content was initially before fertilization happened at Hof2018 (figure 2.21) and Brun2018 (Figure 2.22). There was no significant differences between treatments at individual sampling periods. Calcium content in foliage at Hof2018 was increased one year after fertilization (Figure 2.23), while Brun2018 saw a decrease in calcium content in foliage one year after fertilization (Figure 2.24). Within individual treatments, Mg content was increased in the foliage between years 0 and 1 in treatments 0 kg ha^{-1} Ca and 800 kg ha^{-1} Ca as Calciprill (Appendix D). Ca content in the soil was increased at time stamps 0.5 and one-year, from where it was before fertilization at year 0 within all individual treatments. No differences could be detected between time stamps 0.5 and one-year for any of the treatments. Treatments 0 kg ha^{-1} Ca and 800 kg ha^{-1} Ca Calciprill had an increase in boron content between years 0 and 1.

Treatment 800 kg ha^{-1} Ca as Calciprill also had an increase of zinc in the soil in year 1 (Appendix E).

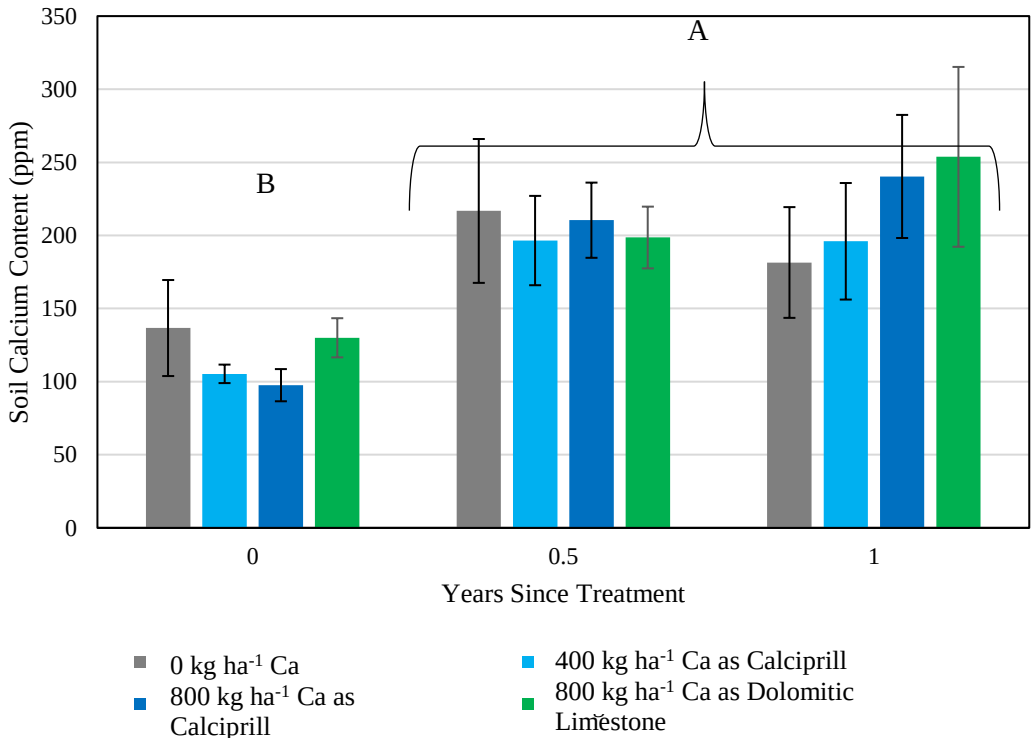


Figure 2.23. Calcium content of soil before treatment, six months after treatment, and one year after treatment from sites Hof2018. Error bars represent standard error of the model. Time stamps compared against each other and statistically significant at $p < 0.0001$.

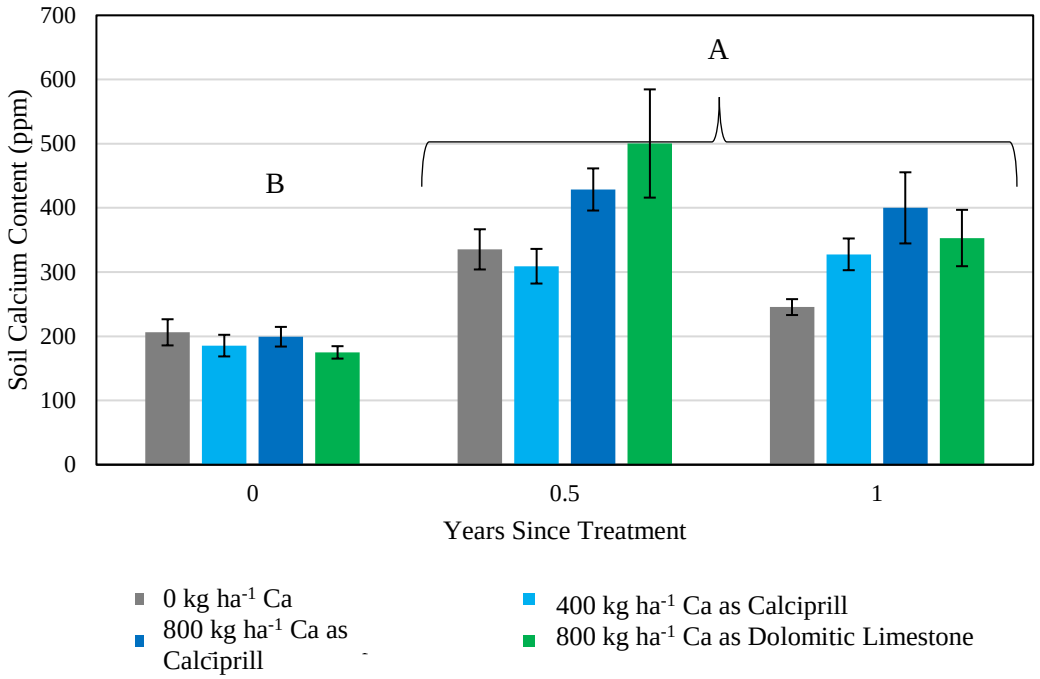


Figure 2.24. Calcium content of soil before treatment, six months after treatment, and one year after treatment from sites Brun2018. Error bars represent standard error of the model.Time stamps compared against each other and statistically significant at $p < 0.0001$.

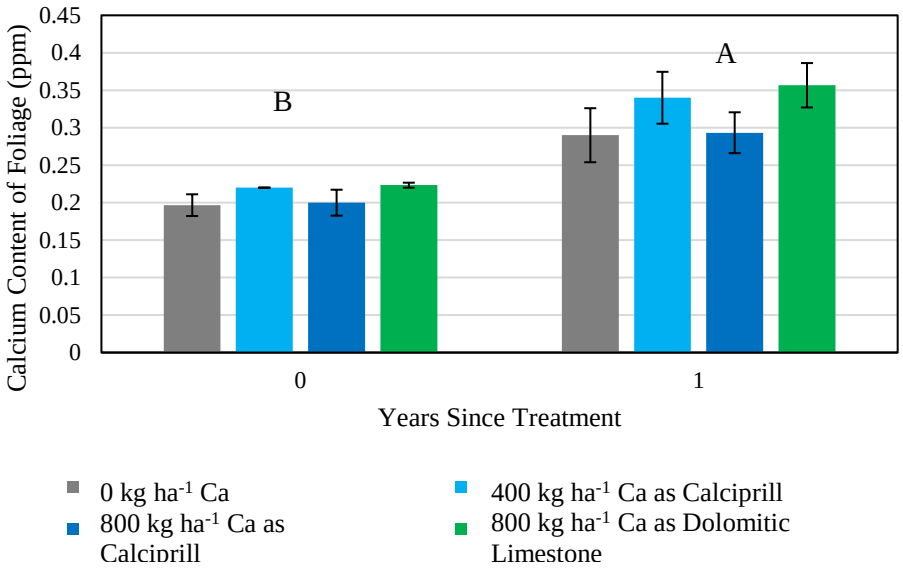


Figure 2.25. Calcium content in foliage before treatment and one year after treatment from site Hof2018 by treatment and YST. Error bars represent standard error of the model. Year 0 and Year 1 differences are significant at $p<0.0001$.

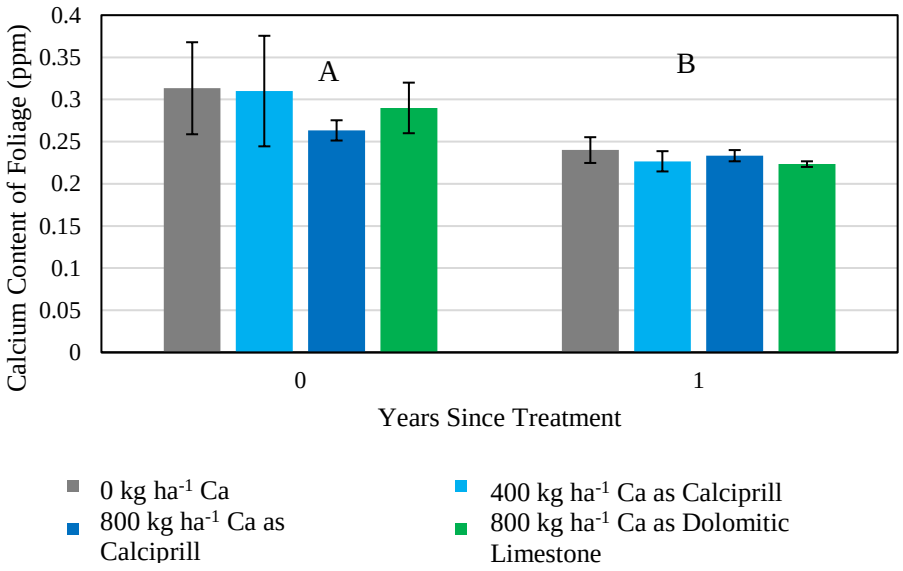


Figure 2.26. Calcium content in foliage before treatment and one year after treatment from site Brun2018 by treatment and YST. Error bars represent standard error of the model. Year 0 and Year 1 differences are significant at $p=0.0013$.

Soil pH Incubation Experiment

Pretreatment pH was 2.61 with the use of $CaCl_2$. The soil pH incubation showed differences in treatments after each incubation period ($p < 0.0001$; Figure 2.25). On day 0 (immediately after treatment), all treatments were statistically different from one another. On day 1, treatments 0 kg ha^{-1} Ca (pH = 2.57) and 400 kg ha^{-1} Ca as Calciprill (pH =2.61) were similar in pH. The 800 kg ha^{-1} Ca a Calciprill pH was 2.86. The 800 kg ha^{-1} Ca as Dolomitic Limestone (pH = 3.06) and 1600 kg ha^{-1} Ca as Calciprill (pH = 3.14) were also similar. Finishing out day 1, 3200 kg ha^{-1} Ca as Calciprill pH was 3.91. On day 2, no difference detected between treatments 0 kg ha^{-1} Ca and 400 kg ha^{-1} Ca as Calciprill. Throughout the rest of the incubation periods, pH maintained the same relative differences with higher levels of application increasing soil pH. The 800 kg ha^{-1} Ca as Dolomitic Limestone treatment consistently remained above the 800 kg ha^{-1} Ca as Calciprill treatment.

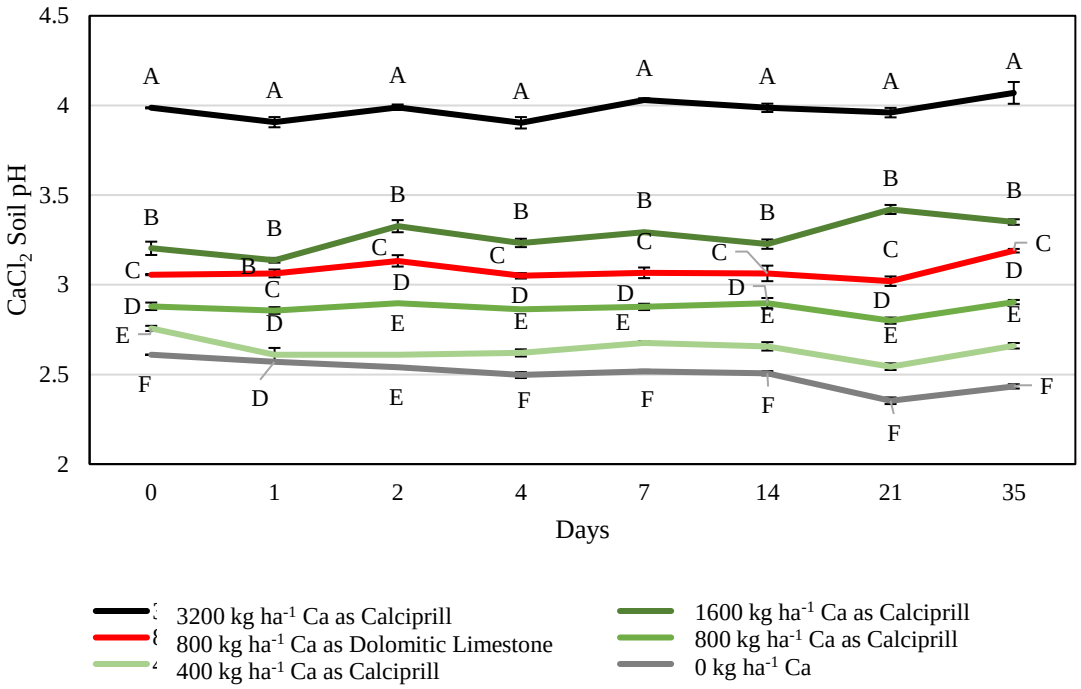


Figure 2.27. Soil pH lab incubation experiment with treatment descriptions and treatment codes. Error bars represent standard error of the model. Significant results at $p < 0.0001$.

Discussion

Mortality

In October 2018, one growing season after treatments had been applied at both study sites, Hurricane Michael laid 90% of treatment trees horizontally on the ground (Figure 2.26). Trees were propped up with stakes and twine, the effort by the rescue crew saved both sites with only a 13% mortality rate between the two sites.

While mortality rate was low, trees likely suffered a setback in growth due to hurricane damage most likely from severing of roots at the base and from flooding. This damage likely delayed treatment separation. Even with efforts to prop trees back to vertical, some trees at Brun2018 are growing with a severe lean. Damaged stems have been shown to have stunted diameter growth compared to undamaged stems for up to six years post-hurricane (Tanner et al. 2014). This could explain why we are seeing greater growth at Hof2018 over Brun2018, even though they are same age, because Brun2018 experienced more damaged than Hof2018.

Figure 2.28. Aftermath of Hurricane Michael, October 2018, at Brun2018.

Tree mortality has continued to tick upward significantly at Hof2018, which is consistent with the findings from Tanner et al. (2014) showing increased mortality in the first six years following a hurricane. Brun2018 experienced its biggest gain of mortality the year directly after the hurricane and has remained at the same level the following years. We think the amount of mortality and damage at this site is masking treatment effect at this site. Additionally, flooding and erosion may have moved some fertilizer off treatment plots.

Calciprill vs Dolomitic Limestone

Calciprill is pure calcium carbonate ($CaCO_3$), whereas dolomitic limestone which 50% $CaCO_3$ and 40% magnesium carbonate (Pagani and Mallarino, 2012). Calciprill that was used in this study was pulverized very finely and then compressed into a small pellet (2-6 mm). The dolomitic limestone used was a very fine loose powder. Calciprill was much easier to distribute than the dolomitic limestone, which lingered in the air and difficult to work with. If sources give similar responses, foresters could choose whichever is cheaper and meets their operational objectives or constraints.

We have supported our hypothesis that there would be no differences in growth response due to Ca sources at the same rate. No growth differences were detected between the 800 kg ha^{-1} Ca treatments in height, DBH, HTLC, or sinuosity rating. Results are similar to the result reported by Pagani and Mallarino (2012), where there were no growth differences between their three different Ca sources.

However, differences among genetics did occur within the same treatments. In treatment 800 kg ha-1 Ca as Calciprill, genetic AGM-22 trees were taller than those of AG-783. In treatment 400 kg ha-1 Ca as Calciprill, genetic AGM-37 trees were taller and had greater DBH than those of AG-783.

Increasing Rates

Height, DBH, HTLC and sinuosity did not differ with increasing rates to 800 kg ha^{-1} Ca treatments over the control at Brun2018. At Hof2018, the 800 kg ha^{-1} Ca treatments did not improve sinuosity over the 400 kg ha^{-1} Ca treatment, and they were comparable to the control. Fox and Jones (2012) found that stems with smaller DBH tended to be less sinuous, while larger DBH stems were more sinuous.

As very little treatment differences were seen between the control and the highest rates of application in height growth or stem form, we hypothesize hurricane damage may have contributed to a lack of treatment response or rates may not have been high enough to elicit a response. MacCarthy and Davey (1976) showed doubled tree growth compared to their control from Ca fertilization on loblolly pines in organic soils at the Hofmann Forest but applied approximately three times higher than our study with 2240 kg ha^{-1} Ca as dolomitic limestone, 380 kg ha^{-1} Ca as calphos, and 2620 kg ha^{-1} Ca as dolomitic limestone + calphos. Similar to our study, the lower rate of only calphos showed growth responses and soil pH similar to their control, leading us to hypothesize that our rates were not high enough to show a treatment response.

MacCarthy and Davey (1976) started with a soil pH of 3.5 and raised it to 4.2 with their dolomitic limestone treatment. Our starting pH at Hof2018 was 3.75, and our 800 kg ha^{-1} Ca as dolomitic limestone treatment raised pH to 3.9, which was statistically similar to our year 0 control. Other studies have used lower or similar rates to those in this study (Espinoza et al. (2012) used 168 kg ha^{-1} Ca as calcium sulfate and Kyle et al. (2005) used 518 kg ha^{-1} Ca as dolomitic limestone) and found treatment effects of improved stem sinousity and height growth, but those studies were not done on organic soil, which has a much larger buffering capacity. The

soil pH at our study sites shifted from 3.75 to 3.9 at Hof2018 and 3.4 to 3.9 at Brun2018 ($p = 0.0187$). The pH at both sites is still below the generally accepted threshold for loblolly pine growth. It may be that our initial treatment rates were too low to see a large treatment effects, that hurricane damaged stems are still recovering, or that Ca is not a limiting nutrient in these systems.

Laboratory Incubation and Field pH

The results from the laboratory incubation experiment showed that the 400 kg ha^{-1} Ca as Calciprill treatment raised soil pH over the control only very slightly and not on all days of the incubation. In the field at Hof2018, all of the treatments soil pH from year one were similar to one another. In the field at Brun2018, the soil pH after one-year post fertilization from the 400 kg ha^{-1} Ca as Calciprill treatment was similar to the control. In the incubation, the 800 kg ha^{-1} Ca as Calciprill treatment was always higher than the 400 kg ha^{-1} treatment, but the effect on soil pH in the field was not significant. We expected treatments under undisturbed laboratory conditions to be more consistent and predictable than results from field conditions where weather and soil disturbance can influence results, and our initial thoughts were supported that the 400 and 800 kg ha^{-1} Ca rates may not have raised pH enough to have an effect on tree growth response. In the field at Hof2018, all of the treatments soil pH from year one were similar to one another, proving that these rates had too many hurdles out in the field to be effective.

While in the field we found no differences among the control, 800 kg ha^{-1} treatments either as dolomitic lime or Calciprill one year after fertilization, we did detect differences in these treatments in the laboratory incubation. This is due to differing amounts and types of CO_3 contents in both products. Calciprill contains 56.6% CO_3 (90.3% $CaCO_3$ (36% Ca) 3.3% $MgCO_3$ (1% Mg)), whereas the amount of CO_3 in the dolomitic lime used was 90.7% (57.7% $CaCO_3$ and

33% $MgCO_3$). We originally hypothesized that the two sources at the same rate of Ca would be similar, but the dolomitic lime raised pH more than the Calciprill. Dolomitic limestone is usually less reactive than pure $CaCO_3$. On the other hand, $MgCO_3$ does have a higher acid neutralizing potential over $CaCO_3$ (Pagani and Mallarino 2012, Adams 1984, Stevens and Blanchar 1992). Jones and Mallarino (2017) found that the more surface area per unit weight a Ca fertilizer has, the quicker it can dissolve and neutralize soil acidity. Calciprill has a higher surface area than the dolomitic limestone used in this study, but in the lab it raised the pH more than its counterpart Calciprill treatment.

We found it took applying the equivalent of 3200 kg ha^{-1} Ca to raised soil pH 1.5 units to reach a pH of 4, or within 0.5 units of the optimum pH range for loblolly pine. MacCarthy and Davey (1976) found that their application of 2240 kg ha^{-1} Ca as dolomitic limestone treatment only raised soil pH 0.7 units. It is possible their source of dolomitic lime was less chemically reactive.

Study Limitations and Future Directions

Several limitations arose during the installment and maintenance including a hurricane and Covid-19 delays. While this research study has two sites matured to three years since treatment, this project is still currently underway and we expect continuing informative results. New sites have been established to address the questions raised in the initial two field studies that include higher rates of application and variations in weed control and establishment P.

Conclusion

Initial results after three years indicate separation of treatment effects on DBH, volume, and stem form, and some interactions with genetics on growth response. While we now know

that the original treatments were not sufficient to raise soil pH to desirable levels for loblolly pine, new study sites with higher application rates should help elucidate any potential rate responses and help us have a better understanding of the impacts Ca fertilization has on acidic organic soil and on the growth and stem form of loblolly pine.

REFERENCES

Adams, F. 1984. Soil acidity and liming (2nd ed.).Agron. Monogr. No. 12. ASA, Madison, WI.

Albaugh, Timothy J, H L Allen, P M Dougherty, and L W Kress. "Leaf Area and Above- and Belowground Growth Responses of Loblolly Pine to Nutrient and Water Additions." *Forest Science* 44, no. 2 (May 1998): 317–28.

Albaugh, Timothy J, T R Fox, R L Cook, and J E Raymond. "Forest Fertilizer Applications in the Southeastern United States from 1969 to 2016." *Forest Science* 65, no. 3 (December 2018). https://doi.org/10.1093/forsci/fxy058.

"ArcGIS for Desktop: 10.6.1, English." Computer software. Redlands, CA: ESRI, 2012.

Birk, Elaine M. "Stem and Branch Form of 20-Year-Old Radiata Pine in Relation to Previous Land Use." *Australian Forestry* 54, no. 1-2 (January 17, 1991): 30–39. https://doi.org/10.1080/00049158.1991.10674554.

Campbell, R K. "Phenotypic Variation and Repeatability of Stem Sinuosity in Douglas Fir." *Northwest Science* 39 (1965): 47–59.

Carlson, C A, T R Fox, H L Allen, T J Albaugh, R A Rubilar, and J L Stape. "Growth Responses of Loblolly Pine in the Southeast United States to Midrotation Applications of Nitrogen, Phosphorus, Potassium, and Micronutrients." *Forest Science* 60, no. 1 (July 11, 2013): 157–69. https://doi.org/https://doi.org/10.5849/forsci.12-158.

Clarkson, D T. "Calcium Transport between Tissues and Its Distribution in the Plant." *Plant, Cell and Environment* 7, no. 6 (1984): 449–56. https://doi.org/10.1111/j.1365-3040.1984.tb01435.x.

Croatan Series. National Cooperative Soil Survey, September 2008. https://soilseries.sc.egov.usda.gov/OSD_Docs/C/CROATAN.html.

Dai, Z, Y Liu, X Wang, and D Zhao. "Changes in PH, CEC and Exchangeable Acidity of Some Forest Soils in Southern China During the Last 32-35 Years." *Water, Air, and Soil Pollution* 108 (1998): 377–90.

Espinoza, J A. "Genetic and Nutritional Effects on Stem Sinuosity in Loblolly Pine." January 22, 2009. https://doi.org/http://www.lib.ncsu.edu/resolver/1840.16/4296.

Espinoza, J.A., H.L. Allen, S.E. McKeand, and P.M. Dougherty. "Stem Sinuosity in Loblolly Pine with Nitrogen and Calcium Additions." *Forest Ecology and Management* 265 (2012): 55–61. https://doi.org/10.1016/j.foreco.2011.10.026.

Ferreira, L E.P., S S Zocchi, and D Baron. “Reconciling the Mitscherlich’s Law of Diminishing Returns with Liebig’s Law of the Minimum. Some Results on Crop Modeling.” *Mathematical Biosciences* 293 (2017): 29–37. https://doi.org/10.1016/j.mbs.2017.08.008.

Fox, T R, H L Allen, T J Albaugh, R Rubilar, and C Carlson. “Tree Nutrition and Forest Fertilization of Pine Plantations in the Southern United States.” *Southern Journal of Applied Forestry* 31 (2007b): 5–11.

Hopmans, P, M Kitching, and G Croatto. “Stem Deformity InPinus Radiata Plantations in South-Eastern Australia.” *Plant and Soil* 175, no. 1 (1995): 31–44. https://doi.org/10.1007/bf02413008.

JMP, Version 15. SAS Institute Inc., Cary, NC, 1989-2021

Jones, P D, and T R Fox. “Stem Sinuosity Changes in Young and Merchantable Stands of Pinus Taeda.” *Forest Products Journal* 62, no. 5 (2012): 354–58. https://doi.org/10.13073/0015-7473-62.5.354.

Jones, J D, and A P Mallarino. “Influence of Source and Particle Size of Agricultural Limestone on Efficiency at Increasing Soil PH.” *Soil Science Society of America*, October 17, 2017, 1–40. https://doi.org/10.31274/etd-180810-4958.

Kyle, K H, T R Fox, L J Andrews, W M Aust, J A Burger, and Gerald H Hansen. “Long-Term Effects of Drainage, Bedding, and Fertilization on Growth of Loblolly Pine (Pinus Taeda L.) in the Coastal Plain of Virginia.” *Southern Journal of Applied Forestry* 29, no. 4 (2005): 205–14. https://doi.org/10.1093/sjaf/29.4.205.

MacCarthy, R., and C. B. Davey. “Nutritional Problems of Pinus Taeda L. (Loblolly Pine) Growing on Pocosin Soil.” *Soil Science Society of America Journal* 40, no. 4 (1976): 582–85. https://doi.org/10.2136/sssaj1976.03615995004000040034x.

Magdoff, F R, and R J Bartlett. “Soil PH Buffering Revisited.” *Soil Science Society of America Journal* 49 (1985): 145–48.

Marschner, H. “Mineral Nutrition of Higher Plants.” *Academic Press, London UK*, 1995. https://doi.org/10.1016/b978-0-12-473542-2.x5000-7.

Pagani, A, and A P Mallarino. “Soil Ph and Crop Grain Yield as Affected by the Source and Rate of Lime.” *Soil Science Society of America Journal* 76, no. 5 (September 12, 2012): 1877–86. https://doi.org/10.2136/sssaj2012.0119.

South, D B, W A Carey, and D A Johnson. “COPPER DEFICIENCY IN PINE PLANTATIONS IN THE GEORGIA COASTAL PLAIN.” *USDA Forest Service*, 2004, 387–90.

Spicer, R, B L Gartner, and R L Darbyshire. “Sinuous Stem Growth in a Douglas-Fir (Pseudotsuga Menziesii) Plantation: Growth Patterns and Wood-Quality Effects.”

Canadian Journal of Forest Research 30, no. 5 (2000): 761–68. https://doi.org/10.1139/x00-024.

Stevens, J J, and R W Blanchar. “Soil PH Gradients NEAR Calcite and Dolomite Particles.” *Soil Science Society of America Journal* 56, no. 3 (1992): 967–72. https://doi.org/10.2136/sssaj1992.03615995005600030047x.

Tanner, E V, F Rodriguez-Sanchez, J R Healey, R J Holdaway, and P J Bellingham. “Long-Term Hurricane Damage Effects on Tropical Forest Tree Growth and Mortality.” *Ecology* 95, no. 10 (2014): 2974–83. https://doi.org/10.1890/13-1801.1.

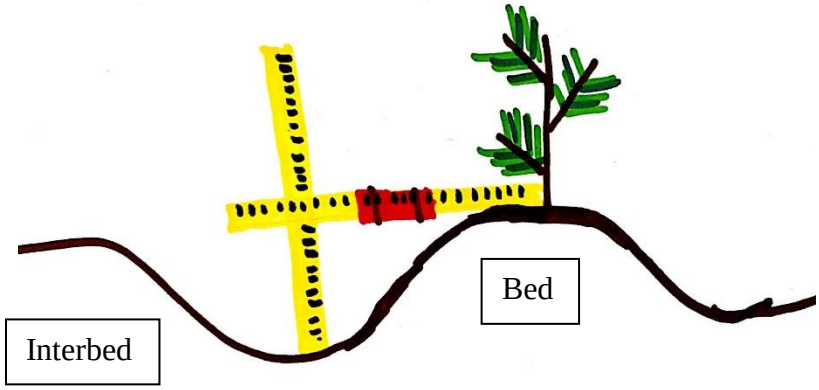
Thompson, W H, C McFarland, T Brown, and D R Huggins. “AGRICULTURAL LIME AND LIMING, PART 1: INTRODUCTION TO AGRICULTURAL LIME AND LIMING.” *Washington State University Extension*, 2011, 1–9.

White, P J, and M R Broadley. “Calcium in Plants.” *Annals of Botany* 92, no. 4 (October 2003): 487–511. https://doi.org/https://doi.org/10.1093/aob/mcg164.

Williams, C G, and C C Lambeth. “Bole Straightness Measurements for Advanced-Generation Loblolly Pine Genetic Tests.” *Silvae Genetica* 38, no. 5-6 (1989): 212–17.

APPENDICES

Appendix A – Bed Height Measurement



Appendix B – Additional Site Installments

The Hofmann site established in 2020 (Hof2020, Figure B1) received higher rates of application than the 2018 trials consisting of a control (no Ca), 800 kg ha^{-1} Ca as Calciprill, 1600 kg ha^{-1} Ca as Calciprill, 3200 kg ha^{-1} Ca as Calciprill, and 0 kg ha^{-1} Ca with operational weed control (whereas all other treatments receive complete weed control). At the Hofmann site established in 2021 (Hof2021, Figure B2) treatments are the same as Hof2020 with an additional treatment of 0 kg ha^{-1} Ca and 0 kg ha^{-1} P to test the relative effect of P additions.

Table B1. Study sites and treatments used at each site with treatment descriptions.

Hof2018 & Brun2018	Hof 2020	Hof2021	Hofmini	Treatment Description
X	X	X	X	0 kg ha^{-1} Ca
X				400 kg ha^{-1} Ca as Calciprill
X	X	X		800 kg ha^{-1} Ca as Calciprill
X				800 kg ha^{-1} Ca as Dolomitic Limestone
	X	X		1600 kg ha^{-1} as Calciprill
	X	X	X	3200 kg ha^{-1} as Calciprill
	X	X		0 kg ha^{-1} Ca with operational weed control
		X		0 kg ha^{-1} Ca and 0 kg ha^{-1} P
			X	3200 kg ha^{-1} Ca as Dolomitic Limestone
			X	6400 kg ha^{-1} Ca as Dolomitic Limestone
			X	12800 kg ha^{-1} Ca as Dolomitic Limestone

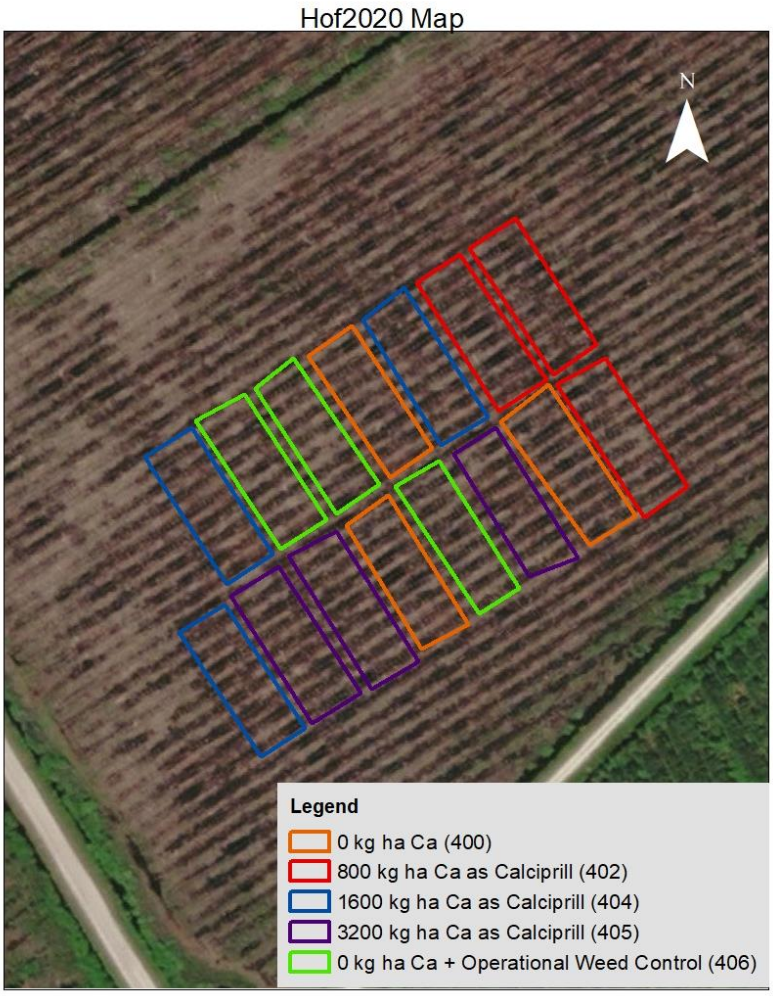


Figure B1. Map of Hof2020 with treatments labeled with treatment descriptions and codes.

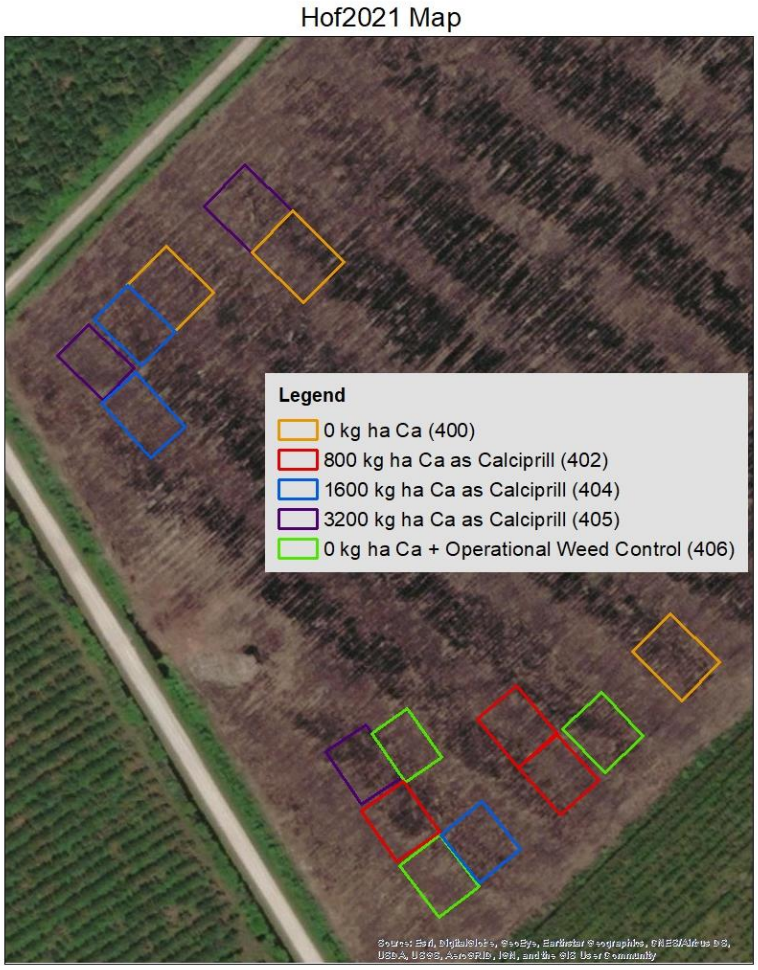


Figure B2. Map of Hof2021 with treatments labeled with treatment descriptions and codes.

In 2020, an additional set of “mini” plots at Hofmann Forest (Hofmini, Figure B3) consisted of five replicates of five treatments with a treatment plot size of three trees (0.0009 ha) in a planting row and one middle tree (tree #2) as measurement tree (Figure B4). The total study area is 0.0225 ha.

Figure B3. Map of Hofmini with treatments labeled with treatment descriptions and codes.

Figure B4. Photo reference of a mini plot.

Appendix C – Sweep

For trees greater than 5 m tall:
Stem straightness will be measured as the maximum deflection (nearest 1 cm) from a meter stick set against the vertical axis of the tree. A straight edge is held next to the tree and moved up along the first long, until the meter stick reaches a height of approximately 5 m. The maximum deflection should be measured in 1 cm increments.

For trees less than 5 m tall:
Stem straightness will be measured as the maximum deflection (nearest 1 cm) from a meter stick set against the vertical axis of the tree up to the height of the tree.
Method based on: Williams 1989

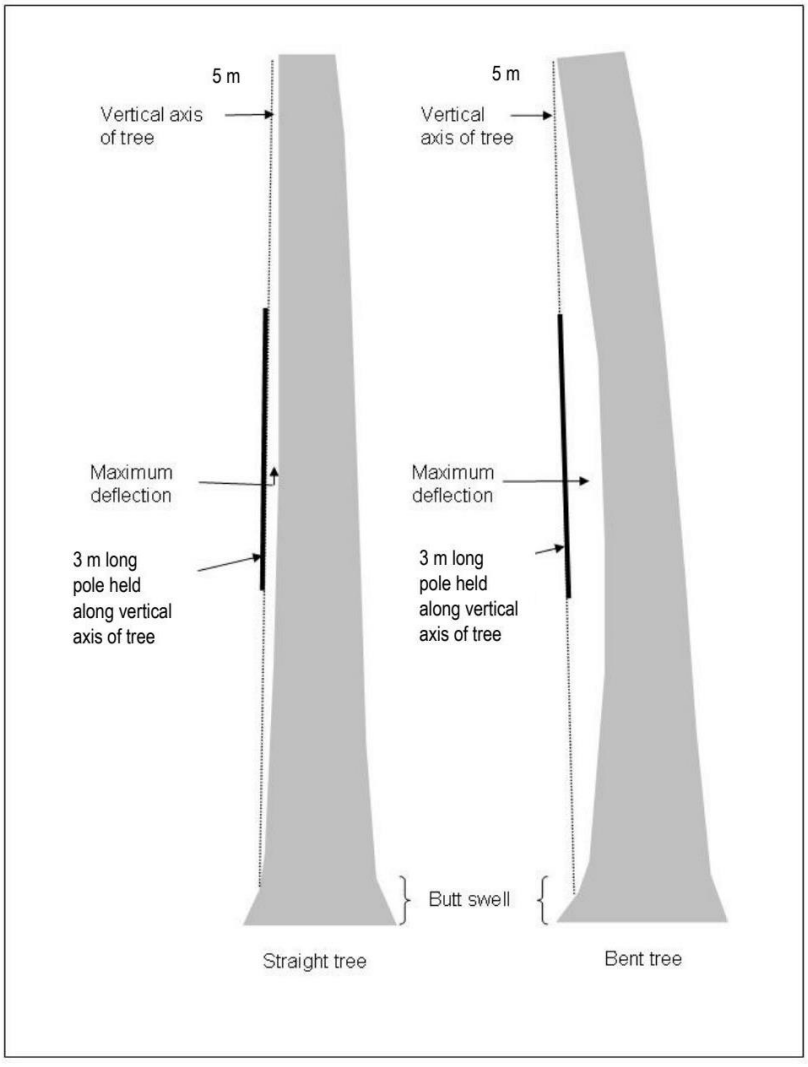


Figure C1. Sweep measurement diagram.

Appendix D – Foliage Nutrient Data

Table D1a. Hof2018 nutrient content data of foliage before fertilization and one year post fertilization by treatment. Statistical significance between year 0 and year 1 separated with letters within individual treatments. No statistical differences between different treatments compared against each other at year 0 or year 1 at p = 0.05

Treatment	YST	N	Ca	P	K	Mg	Mn	S	B	Zn	Cu	Fe
0 kg ha^{-1} Ca	0	1.79	0.19	0.16	0.63	0.07	140.45	0.12	13.85	39.69	1.16	42.39
	1	1.72	0.29	0.16	0.61	0.09	246.62 a	0.12	15.51	40.07	1.27	46.98
400 kg ha^{-1} Ca as Calciprill	0	1.73	0.22	0.15	0.62	0.07	144.6	0.12	14.08	41.55	0.89	59.9
	1	1.66	0.34	0.15	0.62	0.09	237.9	0.12	14.62	40.07	1.07	37.9
800 kg ha^{-1} Ca as Calciprill	0	1.82	0.26	0.16	0.67	0.07	144.39	0.12	13.62	36.01	1.04	42.01
	1	1.71	0.29	0.15	0.58	0.08	250.24 a	0.12	14.79	39.31	1.29	47.34
800 kg ha^{-1} Ca as Dolomitic Limestone	0	1.74	0.22	0.15	0.63	0.07	147.5	0.12	14.43	40.86	1.04	55.34
	1	1.66	0.36	0.15	0.61	0.09	244.8	0.12	14.49	39.01	1.04	55.02

Table D1b Nutrient content data of foliage p-values relating to Table 2.3a.

Hof2018 (yst x treatment)	p-value
N	**0.7844**
Ca	**0.7936**
P	**0.107**
K	**0.8302**
Mg	**0.9303**
Mn	**0.9951**
S	**1.000**
B	**0.6536**
Zn	**0.7713**
Cu	**0.8325**
Fe	**0.3808**

Table D2a. Brun2018 nutrient content data of foliage before fertilization and one year post fertilization by treatment. Statistical significance between year 0 and year 1 separated with letters within individual treatments. No statistical differences between different treatments compared against each other at year 0 or year 1 at p = 0.05.

Treatment	YST	N	Ca	P	K	Mg	Mn	S	B	Zn	Cu	Fe
0 kg ha^{-1} Ca	0	1.41	0.31	0.14	0.59	0.12	141.2	0.11	15.92	33.38	8.07	30.12
	1	1.33	0.24	0.14	0.59	0.12	318.73	0.09	12.37	27.13	2.2	21.03
400 kg ha^{-1} Ca as Calciprill	0	1.39	0.31	0.15	0.64	0.13	139.27	0.11	17.12	36.59	6.37	21.29
	1	1.44	0.23	0.13	0.56	0.11	316.63	0.08	11.33	23.62	1.61	20.48
800 kg ha^{-1} Ca as Calciprill	0	1.41	0.26	0.14	0.57	0.12	143.63	0.1	14.82	32.68	3.23	22.92
	1	1.47	0.23	0.14	0.59	0.12	337.63	0.09	12.93	23.94	1.93	21.38
800 kg ha^{-1} Ca as Dolomitic Limestone	0	1.4	0.29	0.14	0.57	0.12	143.07	0.1	15.38	32.68	5.31	25.11
	1	1.38	0.22	0.13	0.59	0.12	335.49	0.08	12.94	23.24	1.49	21.91

Table D2b. Nutrient content data of foliage p-values relating to Table 2.4a.

Brun2018 (yst x treatment)	p-value
N	**0.9427**
Ca	**0.8622**
P	**0.4424**
K	**0.3836**
Mg	**0.8014**
Mn	**0.5799**
S	**0.4991**
B	**0.6624**
Zn	**0.7153**
Cu	**0.8373**
Fe	**0.4949**

Appendix E – Soil Nutrient Data

Table E1a. Nutrient content data of soil before fertilization for Hof2018, 0.5 year, and one year post fertilization by treatment. Statistical significance between year 0 and year 1 separated with letters within individual treatments at $p \leq 0.05$. No statistical differences were detected among treatments for any time period.

Treatment	YST	P	K	Ca	Mg	Mn	S	B	Z	Cu	Fe
0 kg ha^{-1} Ca	**0**	22.33	20.67	136.67	21.5	139.69	9.23b	0.13	0.43	0.1	32.8
	0.5	42.08	24.92	216.75	47.67						
	1	37.08	26.08	181.5	44.42	174.5	9.89ab	0.15	0.945	0.11	45.25
400 kg ha^{-1} Ca as Calciprill	**0**	23.17	24.83	105.33	27.08	149.87	9.47ab	0.13	0.59	0.1	34.08
	0.5	39.08	26	196.5	48.58						
	1	38.08	28.58	196	46.92	159.17	9.47ab	0.15	0.95	0.11	46.17
800 kg ha^{-1} Ca as Calciprill	**0**	25.25	25	97.58	23.75	145.14	10.61a	0.13	0.5	0.1	34.92
	0.5	37.42	25.17	210.42	46.25						
	1	41.92	26.08	240.33	43.67	162.85	8.85b	0.14	0.93	0.11	41.42
800 kg ha^{-1} Ca as Dolomitic Limestone	**0**	25.17	24.75	130	25.5	151.94	9.48ab	0.14	0.53	0.1	35.33
	0.5	36	22.97	198.58	42.08						
	1	40.25	24.75	253.75	43.25	154.11	9.15b	0.14	0.79	0.14	44.5

Table E1b. Nutrient content data of soil p-values relating to Table 2.5a.

Hof2018 (YSTxTreatment)	P-value
P	0.8062
K	0.8647
Ca	0.7949
Mg	0.9972
Mn	0.9535
S	0.0013
B	0.6247
Zn	0.9366
Cu	0.8665
Fe	0.6417

Table E2a. Nutrient content data of soil before fertilization for Brun2018, 0.5 year, and one year post fertilization by treatment. Statistical significance between year 0 and year 1 separated with letters within individual treatments at $p \leq 0.05$. No statistical differences were detected among treatments for any time period.

Treatment	YST	P	K	Ca	Mg	Mn	S	B	Zn	Cu	Fe
0 kg ha^{-1} Ca	**0**	8.83	38.17	206.17d	117.58	1.75	5.87	0.14	0.79	0.1	62.42
	0.5	16.08	38.92	335.42abcd	144.75						
	1	8.33	34.33	245.5cd	134.5	2	4.94	0.16	0.89	0.11	66.58
400 kg ha^{-1} Ca as Calciprill	**0**	14.08	31.5	185.5d	131.5	2.67	6.19	0.15	0.82	0.15	75.67
	0.5	20.58	31.75	309.17bcd	135.42						
	1	17.58	29.33	327.67abcd	120.08	1.58	5.53	0.19	0.91	0.11	83.58
800 kg ha^{-1} Ca as Calciprill	**0**	7.17	42.5	199.33d	114.5	1.83	5.61	0.14	0.84	0.1	56.58
	0.5	11.08	42.25	428.75ab	143						
	1	8.17	32.33	400.08abc	123.75	2.5	4.94	0.18	1.15	0.17	68.58
800 kg ha^{-1} Ca as Dolomitic Limestone	**0**	9	36.08	174.92d	114.08	2.17	5.78	0.14	0.86	0.11	61.83
	0.5	8.33	37.08	500.33a	148.58						
	1	7.58	32.92	353.08abcd	122.08	2.25	5.19	0.18	1.09	0.18	68.75

Table E2b. Nutrient content data of soil p-values relating to Table 2.5a.

Brun2018 (YSTxTreatment)	P-value
P	0.974
K	0.9112
Ca	0.0434
Mg	0.0942
Mn	0.5021
S	0.9246
B	0.8799
Zn	0.7327
Cu	0.3006
Fe	0.9611